SCC Initiation Research on Alloy 600 and Alloy 690: PNNL Presentations at the EPRI Alloy 690 Expert Meeting

Milestone: M3LW-16OR0402032

Steve Bruemmer, Ziqing Zhai and Mychailo Toloczko

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Milestone Submission

December 11, 2015

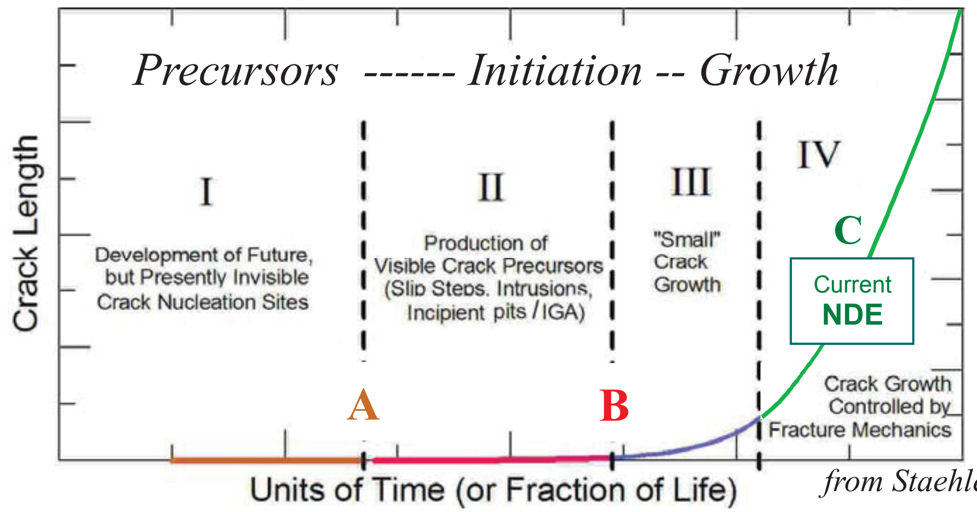
Light Water Reactor Sustainability R&D Program

Overall Project Objectives and Approach

- *Focus is to identify mechanisms controlling SCC initiation of corrosion-resistant nickel-base alloys under realistic LWR service conditions.*
- *Research is investigating important material (composition, microstructure and surface condition) and environmental (water chemistry, temperature and electrochemical potential) effects on the susceptibility to localized corrosion/oxidation and SCC initiation.*
- *High-temperature autoclave test systems have been designed and constructed to enable evaluation of corrosion precursors and in-situ measurement of crack initiation in simulated LWR environments.*
- *Experiments are evaluating surface corrosion-oxidation and SCC crack initiation in alloy 600 & 690 materials with known SCC susceptibility in PWR primary water.*

Background: Nano-to-Microscale SCC Initiation Precursors in LWR Service Environments

- *The evolution of stress-corrosion crack initiation may take decades in service to form, but is often followed by rapid crack growth and component failure.*
- *It is essential to detect early near-surface degradation precursors (**A**) and monitor development to crack initiation (**B**) enabling proactive mitigation before the rapid growth stage (**C**) is reached.*
- ***A key aspect of this research is on the characterization of localized damage, oxidation and crack precursors at the nano-to-microscale in LWR component alloys.***
- *Techniques include high-resolution scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atom probe tomography (APT).*



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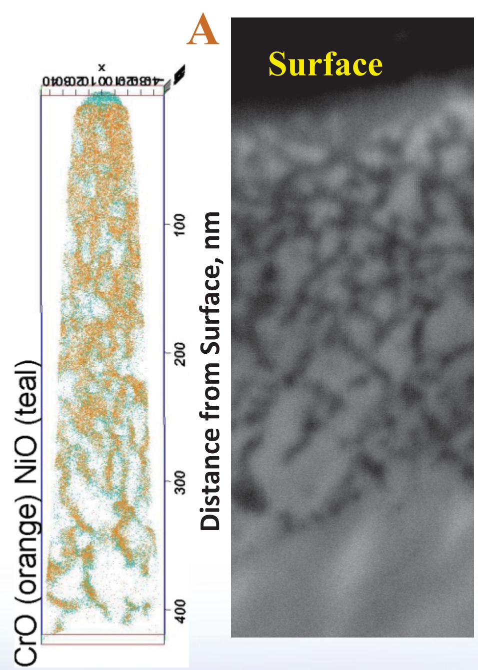
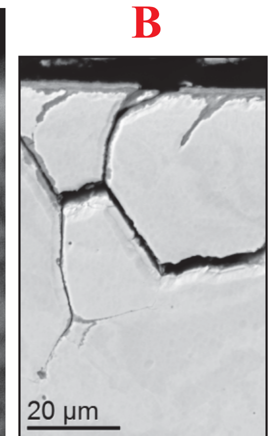
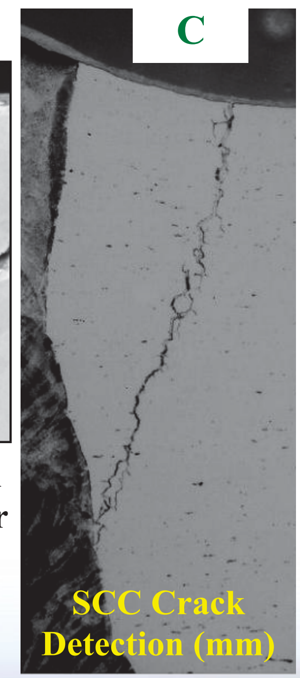


Precursor Damage Penetrative Oxidation (0.1-20 µm)



SCC Initiation from Precursor Damage (10-50 µm)



PNNL Presentations at the EPRI Alloy 690 Expert Meeting: December 1-3, 2015

- ***SCC Crack Initiation in Alloy 600 (Mychailo Toloczko)***
  - ***Reviewed cold work and applied stress effects on SCC initiation times with a focus on quantitative measurement and analysis of crack shape evolution. Results are suggesting a continous cracking process that increases in rate as crack depth and corresponding stress intensity increase. Concurrent crack growth rate tests are determining SCC response at low stress intensities to establish transition behavior during SCC initiation.***
  - ***PNNL research is in excellent agreement with prior work at KAPL and EdF. Results are forming the basis for a multi-laboratory, international round robin on SCC initiation testing of cold-worked alloy 600.***
- ***SCC Initiation Research on Cold-Worked Alloy 690 (Steve Bruemmer)***
  - ***Reviewed blunt-notch and constant load initiation experiments of cold-worked alloy 690 materials susceptible to SCC crack growth.***
  - ***Blunt notch tests ongoing on 21 and 31% cold-forged CRDM materials. Formation of grain boundary creep cavities discovered during dynamic straining promotes SCC initiation in blunt notch tests.***
  - ***Long-term, constant load experiments on cold-worked alloy 690 materials have now reached 1.1 year. High-resolution surface characterizations revealed IG crack nucleation in highly cold-worked CRDM materials due to grain boundary cavity precursors, but no transition to rapid crack growth.***
  - ***First observations of crack nucleation in PWR primary water for alloy 690, considerable discussion occurred on implications for service components.***

Stress Corrosion Crack Initiation of Alloy 600

Mychailo Toloczko, Ziqing Zhai, Matt Olszta and Steve Bruemmer

Pacific Northwest National Laboratory

EPRI Alloy 690/52/152 PWSCC
Research Collaboration Meeting
Tampa, Florida December 1-3, 2015

Light Water Reactor Sustainability

Light Water Reactor Sustainability R&D Program

Background

- *Focus is to identify mechanisms controlling SCC initiation of nickel-base alloys under realistic LWR service conditions.*
- *Investigating the susceptibility to localized corrosion/ oxidation and SCC initiation as a function of:*
  - *Material (composition, microstructure and surface condition)*
  - *Environment (stress, water chemistry, temperature and electrochemical potential)*
- *Using high-temperature autoclave test systems and in-situ measurement of crack initiation under well controlled loading and strains.*
- *Evaluating surface corrosion-oxidation and SCC crack initiation in alloy 600 & 690 materials with known SCC susceptibility in PWR primary water.*
- ***Alloy 600 SCC initiation response versus cold work in this presentation.***

Presentation Outline

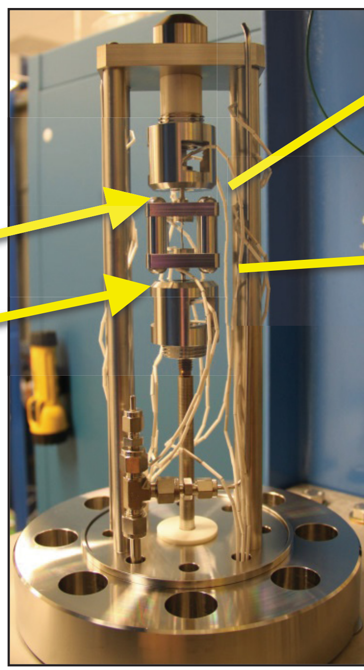
- ***Experimental Setup and Materials***
- *SCC initiation response versus time*
- *SCC initiation versus yield stress and plastic strain*
- *Crack characterization studies*
- *Stress intensity estimates*
- *Low K SCC crack growth rate testing*

SCC Initiation Test Systems

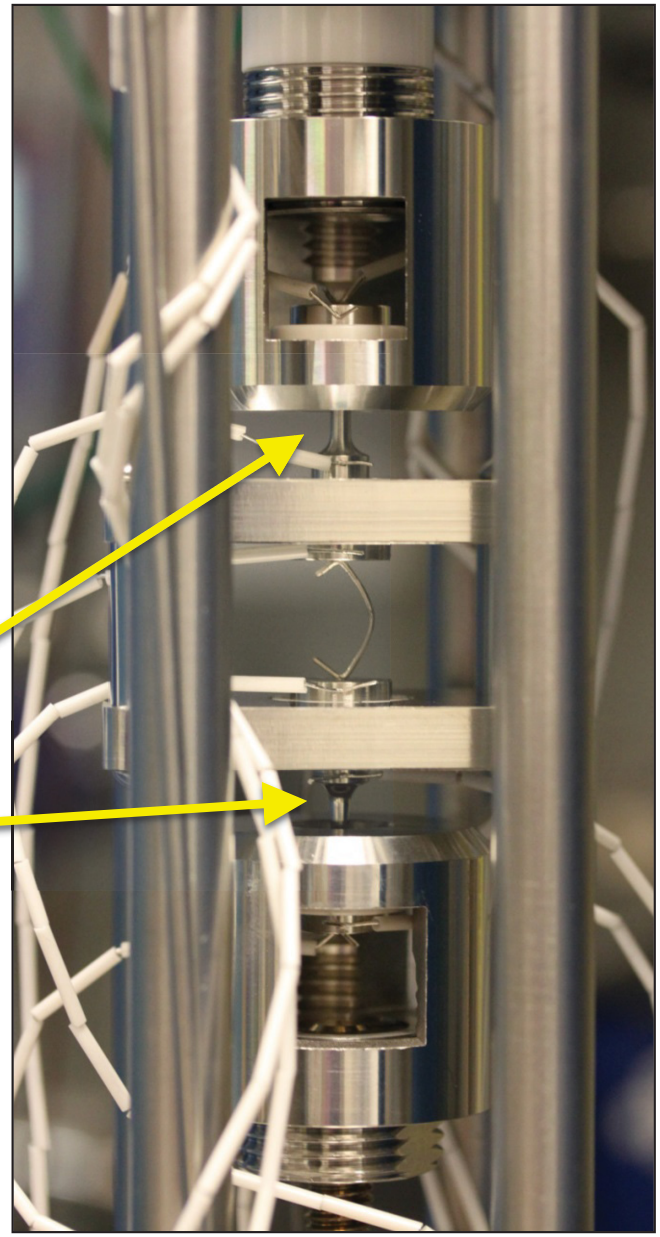
- *Based on PNNL SCC CGR test systems.*
- *Two systems that can test up to 6 specimens.*
- *One 36 specimen system with up to 20 specimens instrumented for initiation.*
- *All use DCPD for in-situ detection of crack initation.*

1.2" Tall SCC Initiation Specimen

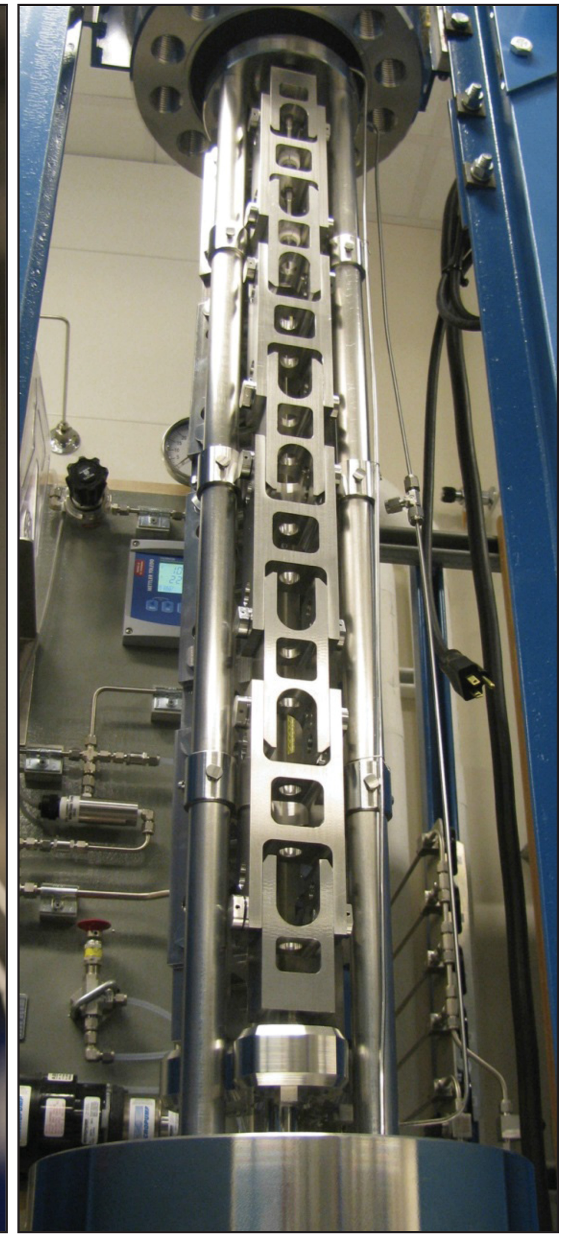
2-6 Specimen SCC Initiation System



2-6 Specimen SCC Initiation System



36 Specimen SCC Initiation System

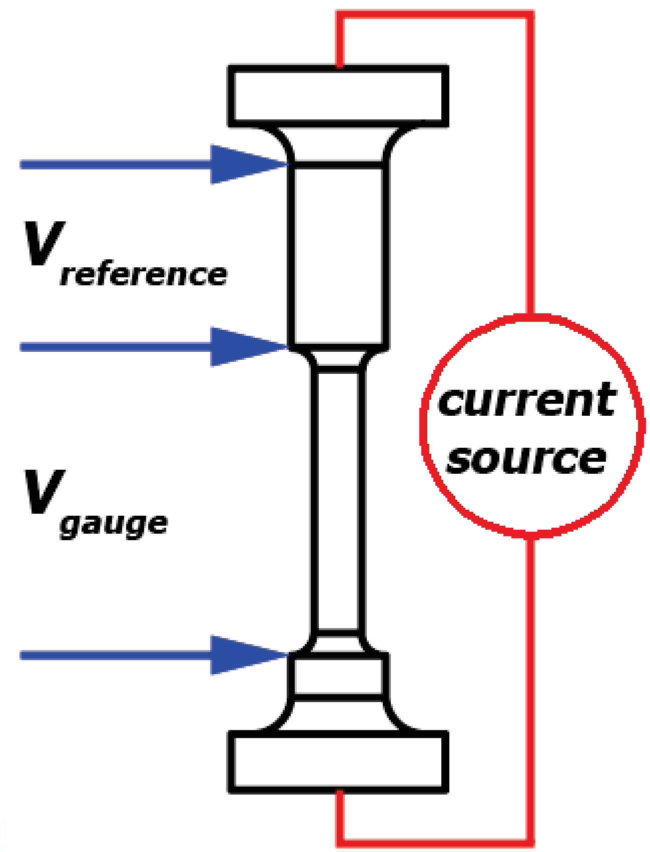


DCPD to Measure Crack Initiation

- *Voltage is sensitive to many factors*
  - *Cracking*
  - *Length and diameter change, e.g, creep and tensile straining*
  - *Material resistivity evolution*
- *Resistivity evolution can be removed, but creep cannot be deconvoluted from the response.*
- *Currently interpreting DCPD response as creep because it has been the dominant effect on DCPD up to initiation.*

$$\varepsilon_{referenced} = \frac{1}{2}\left[\ln\left(\frac{V_{gauge}}{V_{gauge_o}}\right) - \ln\left(\frac{V_{ref}}{V_{ref_o}}\right)\right]$$

Schematic of Idealized Voltage Measurement Points

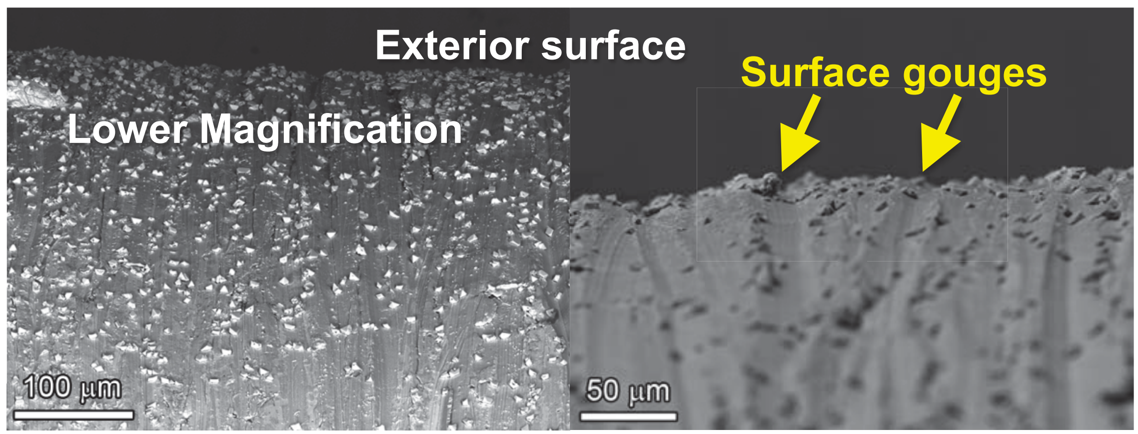


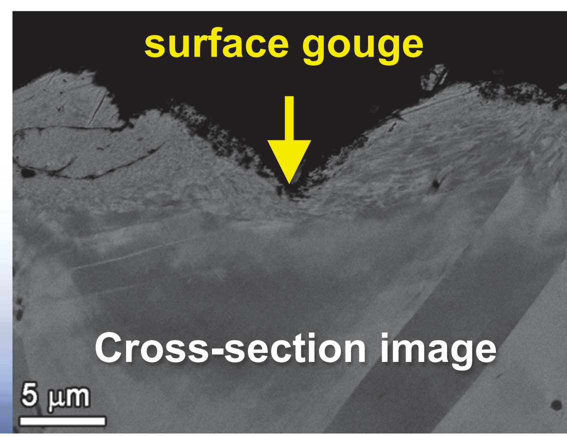
Light Water Reactor Sustainability

LWSR Alloy 600 Materials

- *Three different heats of material – 1 plate and 2 CRDM.*
- *All materials started off in their as-received, mill annealed condition.*
- *0 – 20% CW specimens were generated, cold work achieved by rolling or tensile straining at room temperature.*
- *After cold working, gauge region was prepared with either a 1 µm surface finish or a ground finish. Applied with a lathe.*

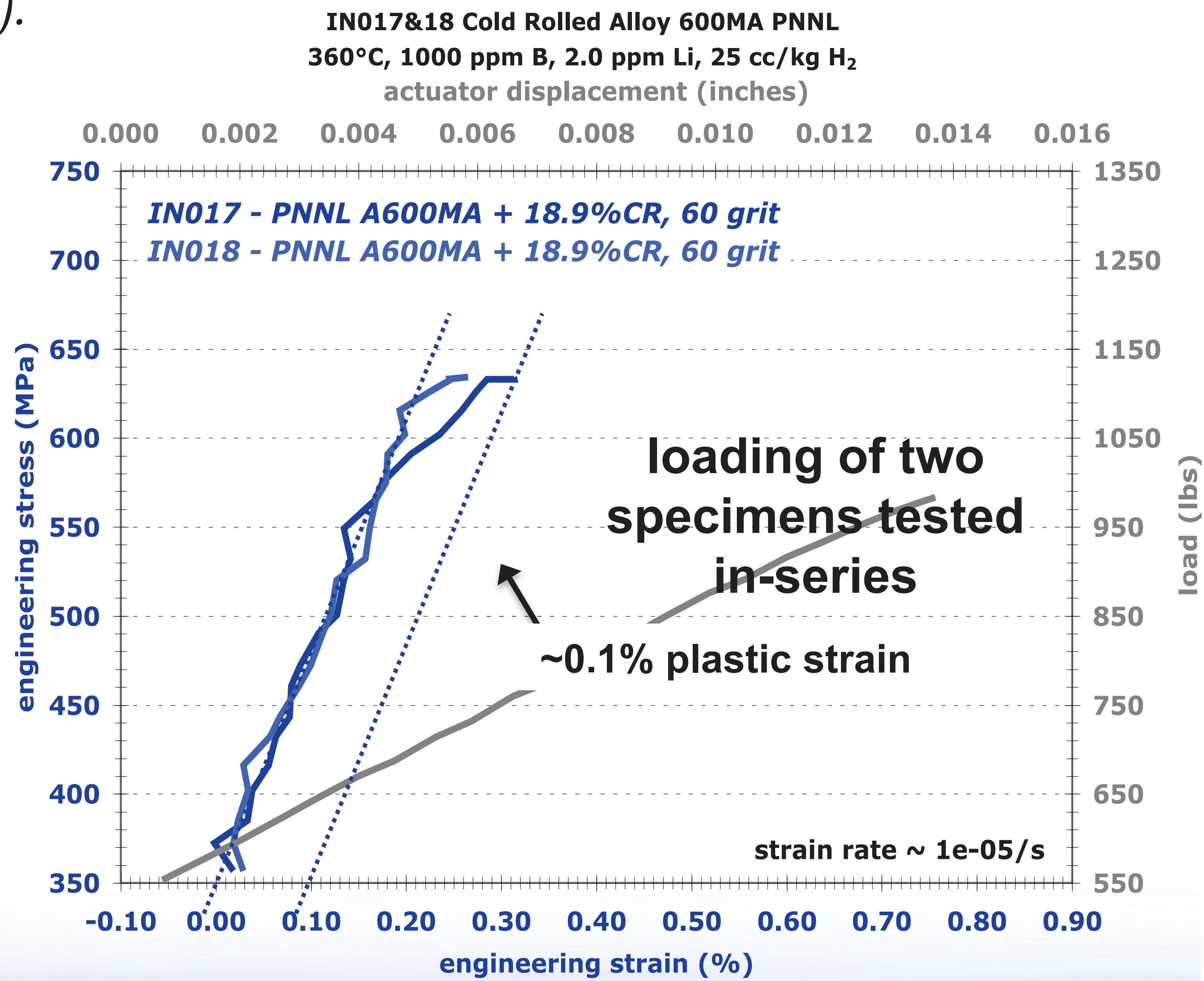
Example shown is a 60 grit ground finish to approximate typical surface condition in LWR service.





Test Conditions

- *PWR primary water – 1000 ppm B, 2 ppm Li, 360°C, 25 cc/kg H_2O (Ni/NiO stability line).*
- *All specimens loaded to their yield strength.*
- *Yield observed by monitoring stress (from load) versus strain (from DCPD) at a displacement rate of ~$1x10^{-5}$ s^{-1} (~1 hour to load).*
- *For multi-specimen tests, goal was to limit plastic strain during loading to <1.5%.*



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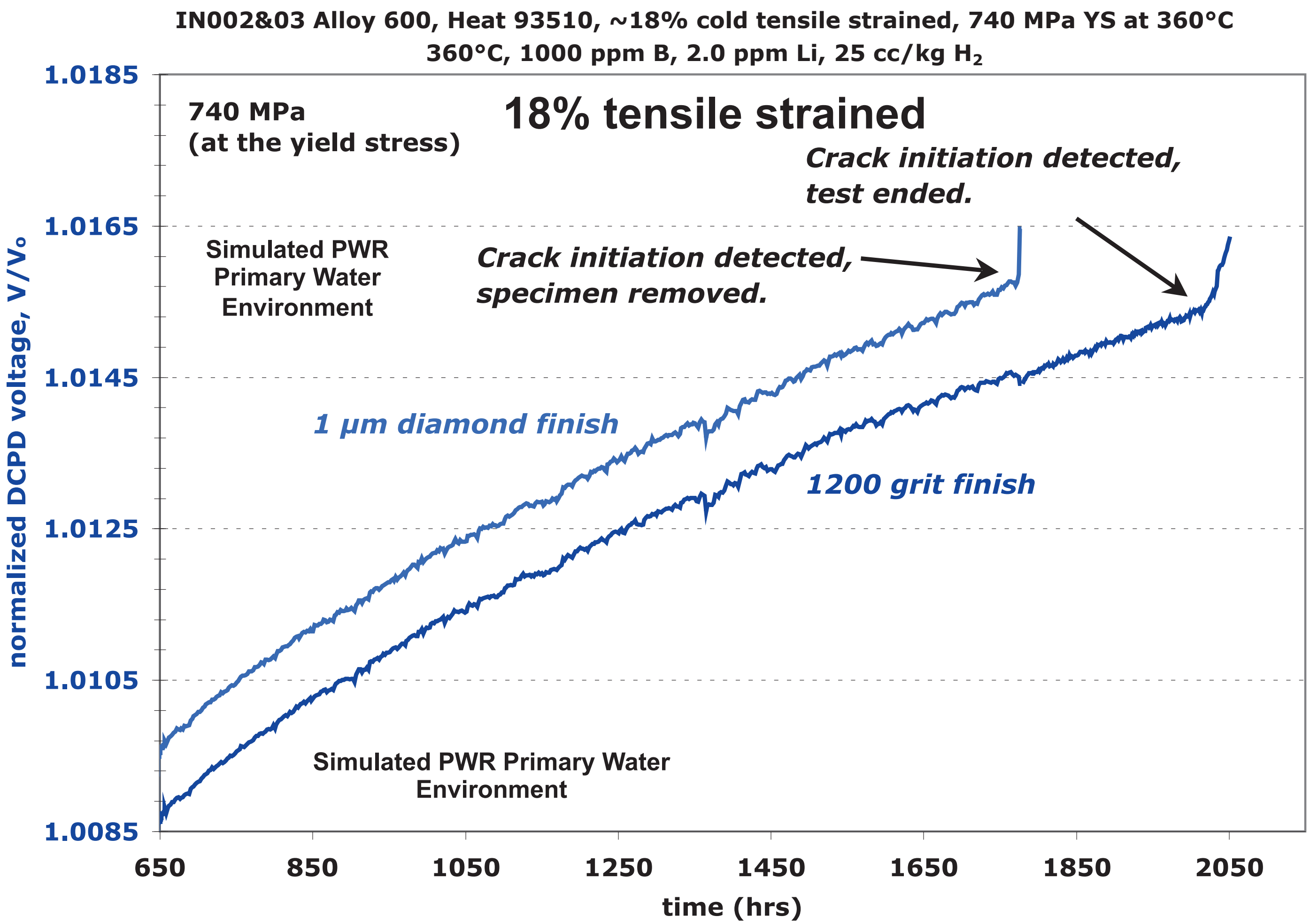
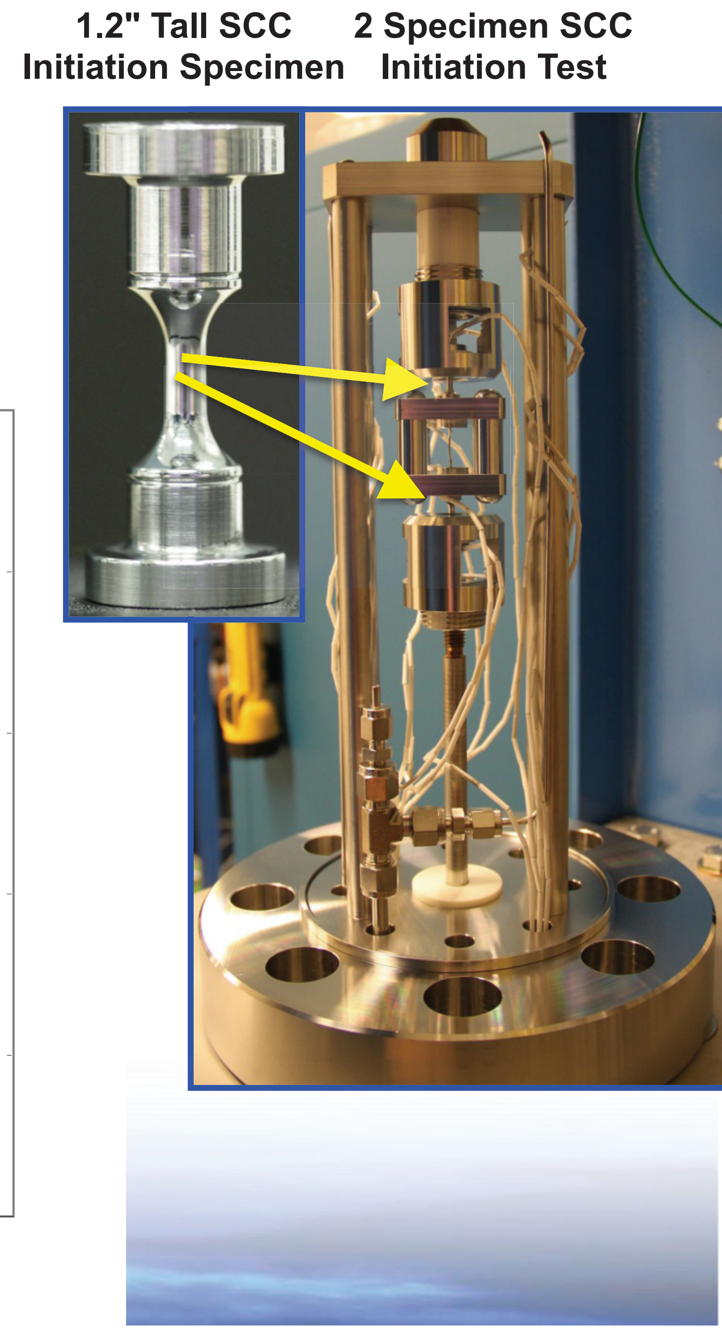
Presentation Outline

- *Experimental Setup and Materials*
- ***SCC initiation response versus time***
- *SCC initiation versus yield stress and plastic strain*
- *Crack characterization studies*
- *Stress intensity estimates*
- *Low K SCC crack growth rate testing*

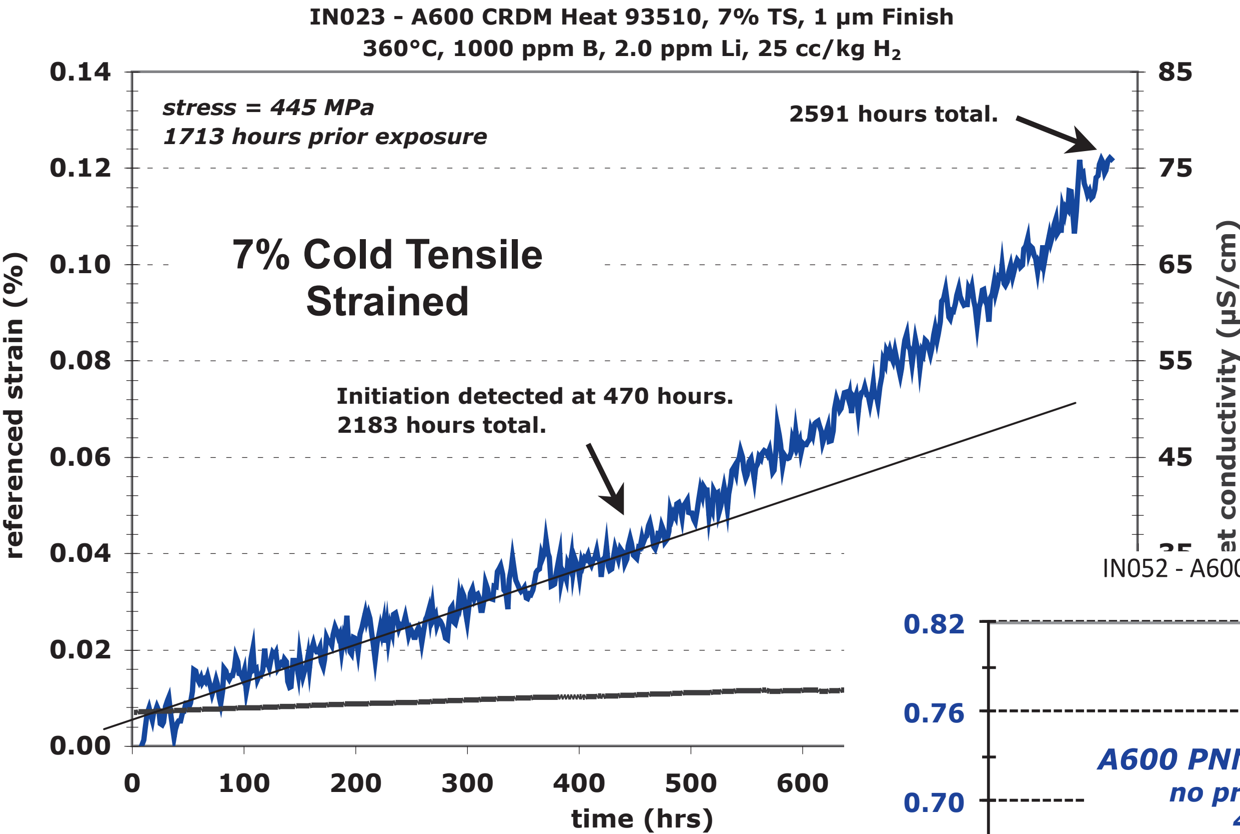
Initiation Response of Cold-Worked Materials

- *Highly cold worked material show a smooth but rapid transition to a higher indicated DCPD or strain slope.*



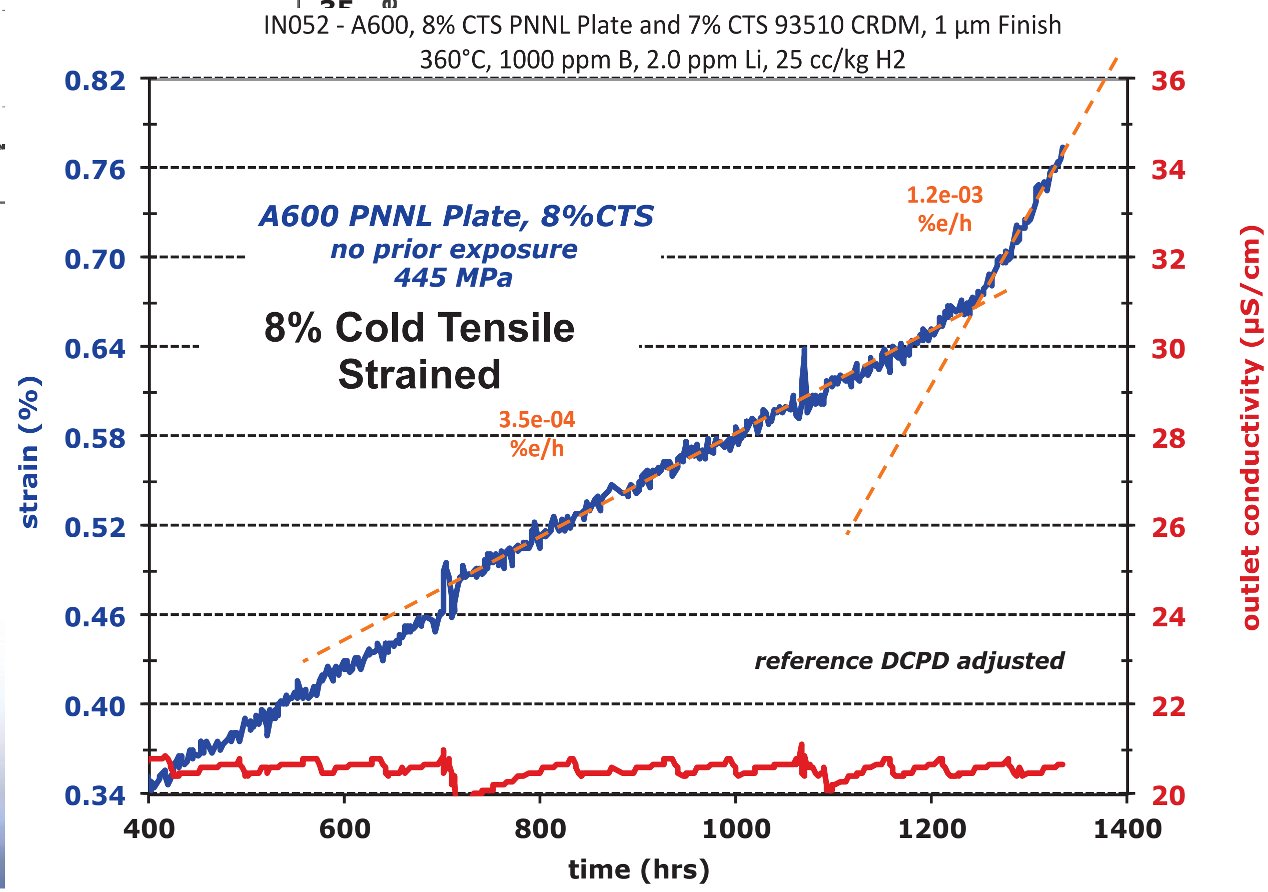


Response of Moderately Cold Worked Materials

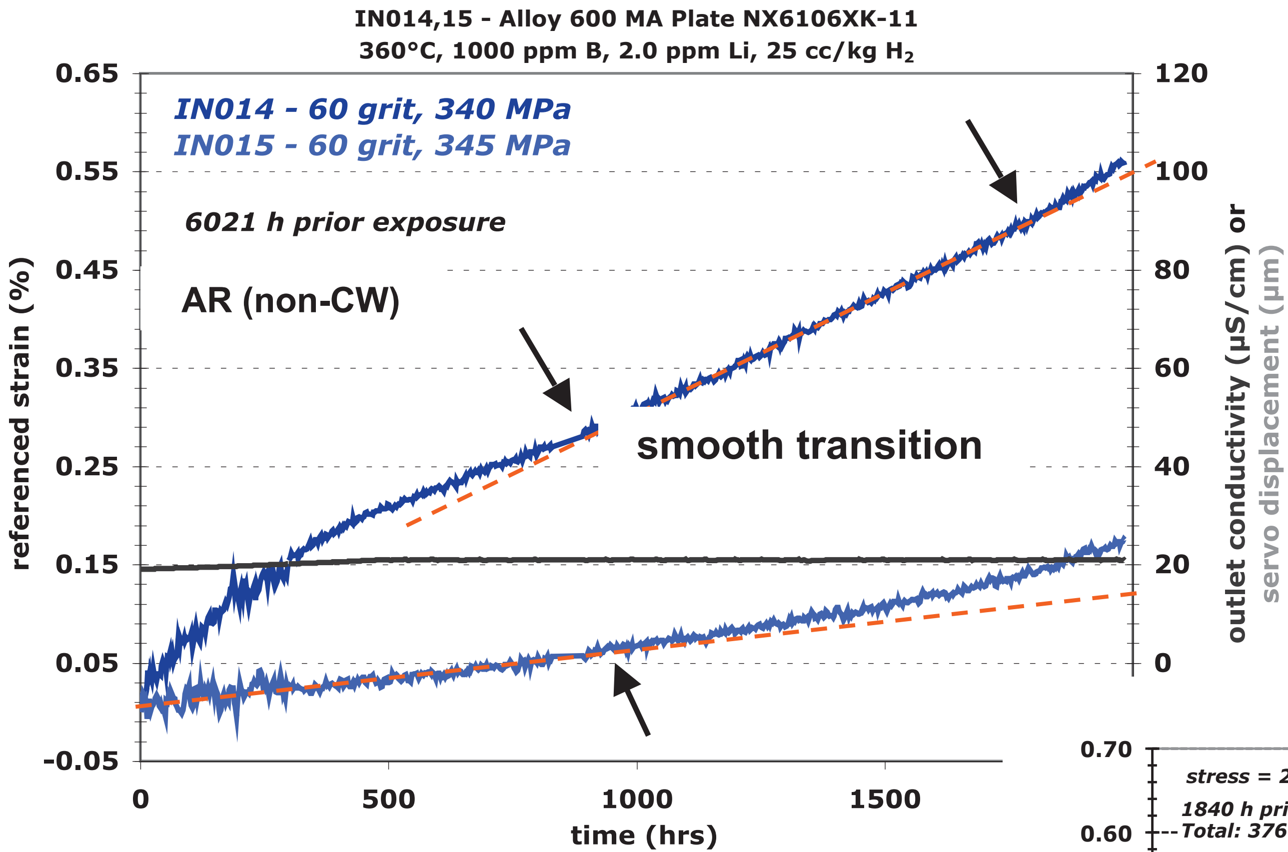


Materials cold worked to "moderate" levels are exhibiting a longer transition in DCPD response.

Different response is likely due to differences in SCC susceptibility and applied stress.

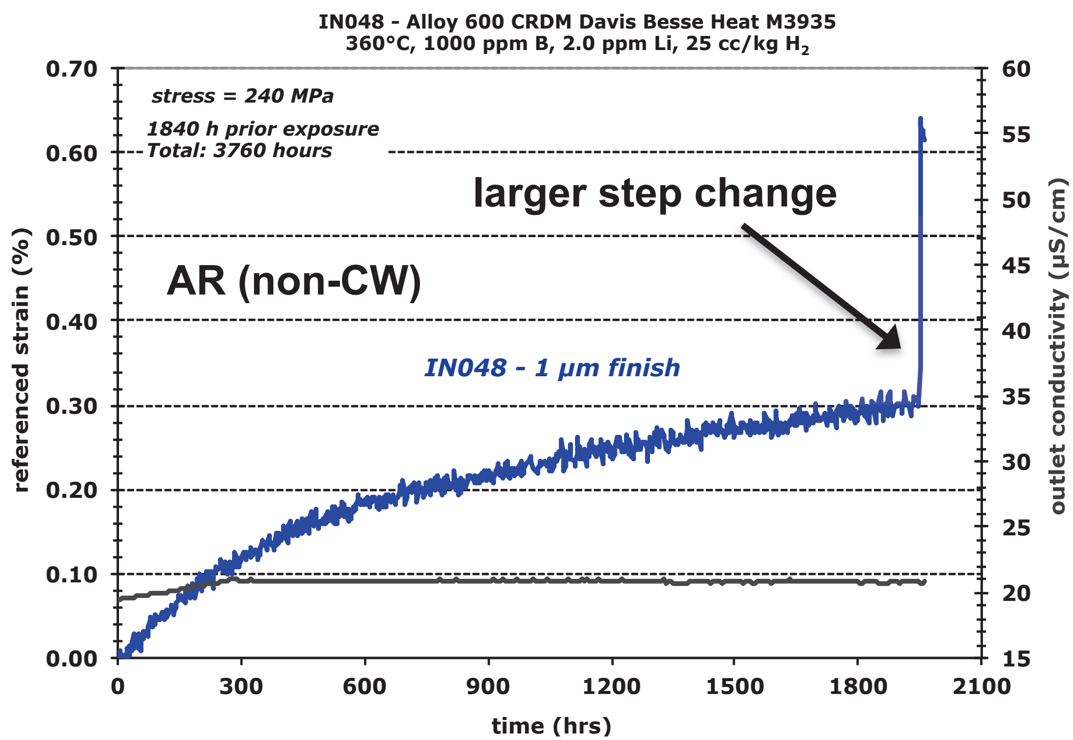


Non-Cold Worked Materials



For non-CW materials, some specimens have exhibited a smooth transition to higher slope. Other materials have exhibited a step-change in strain.

Testing continues on various as-received, non-CW alloy 690 materials to better understand response.



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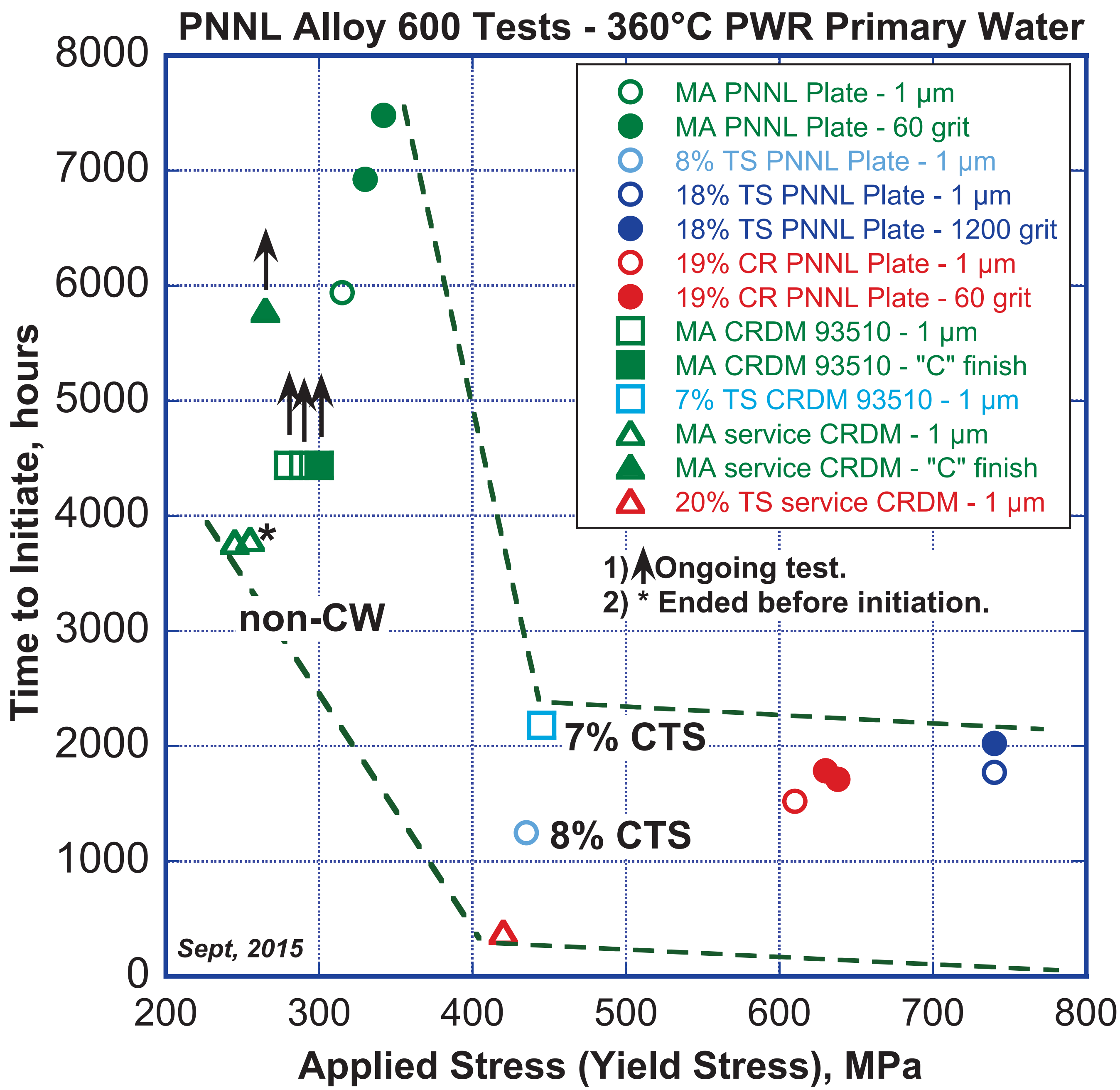
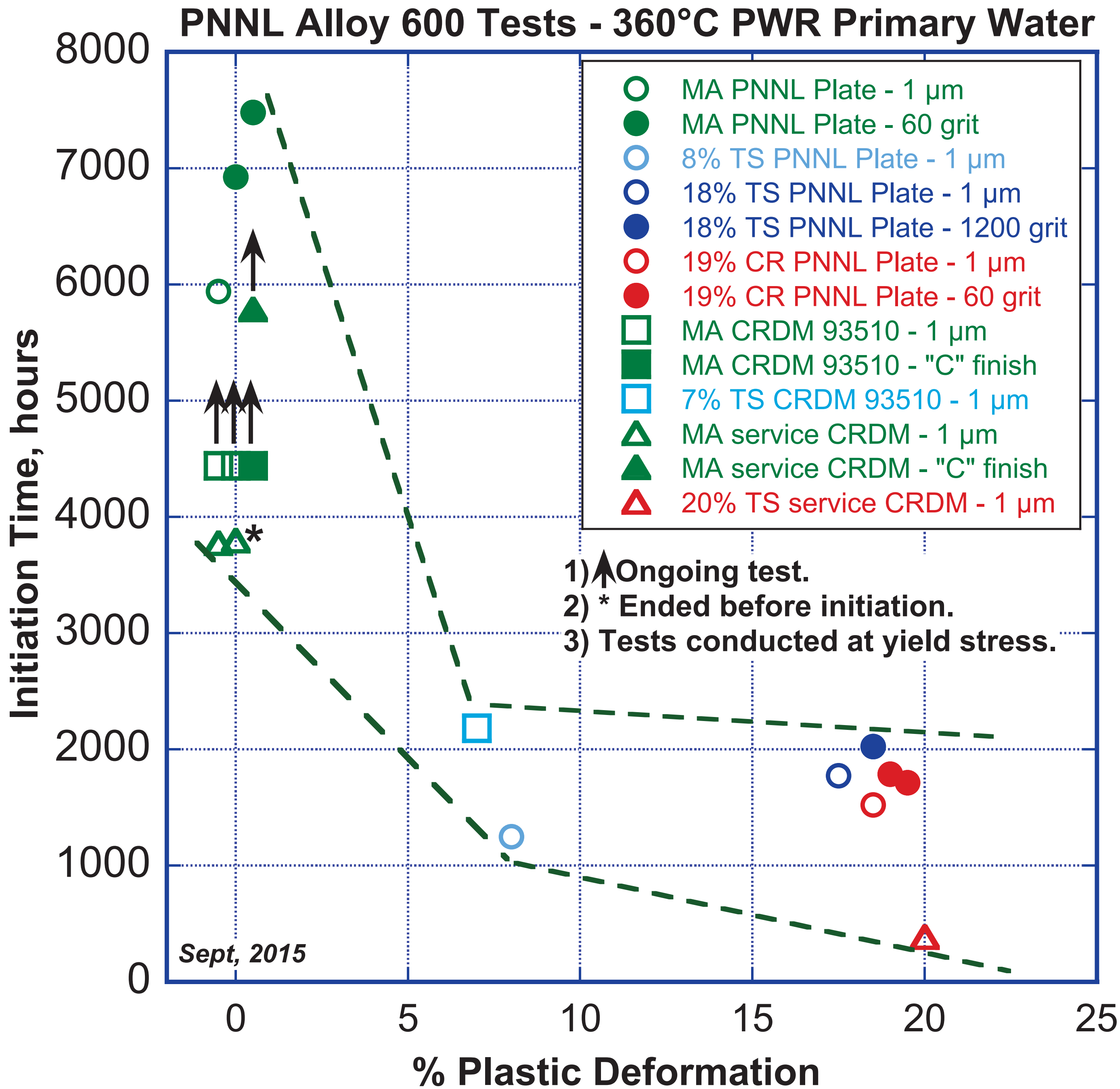
SCC Initiation Response Versus Yield Strength

- *All specimens were at constant applied load equal to material YS.*
- *Possible initiation of two heats in non-CW condition.*
- *7-8% CW induced a strong reduction in initiation time.*
- *Service CRDM (large grain size, high B content) exhibiting low initiation times.*



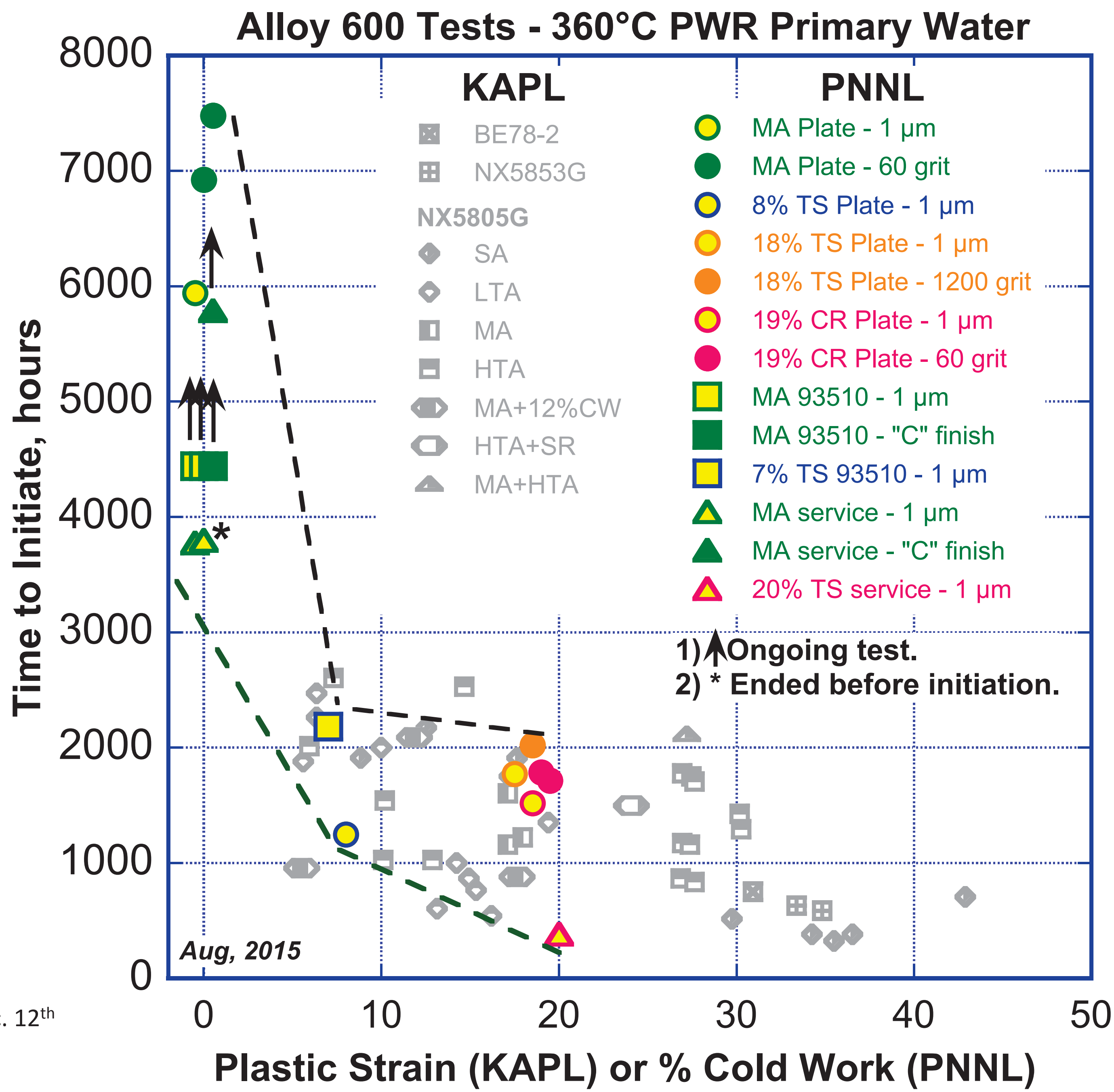
SCC Initiation Response Versus Cold Work

- *Very similar trend when plotted versus cold work.*
- *More tests are needed to confirm and fill out the response as a function of cold work and applied stress.*



Comparison of PNNL Alloy 600 Results to KAPL

- *PNNL data agrees well with KAPL data when plotted versus cold work (PNNL) or plastic strain (KAPL).*



KAPL data from: Richey, Morton and Schurman, Proc. 12th Int. Conf. Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors

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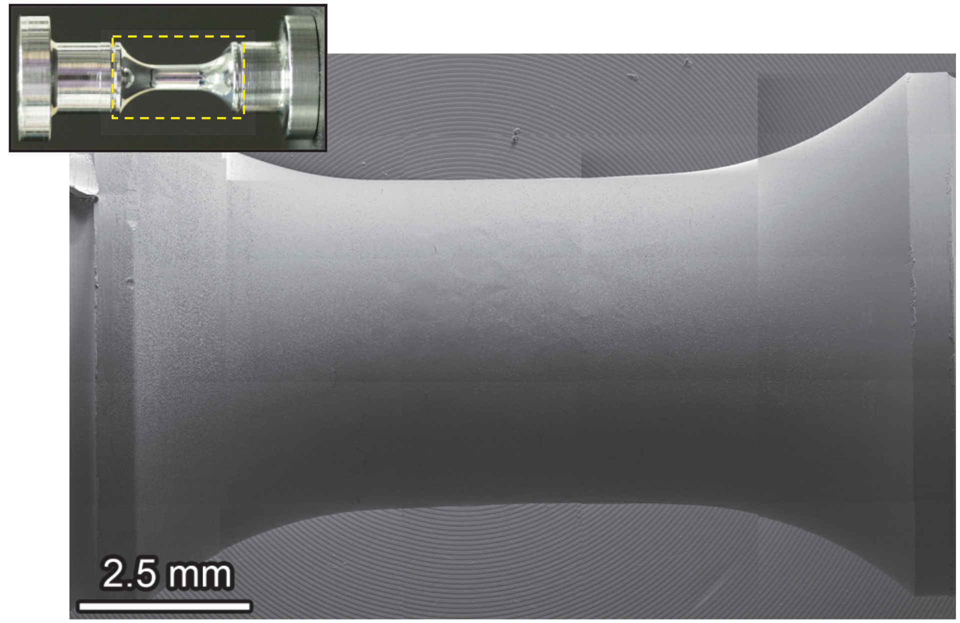
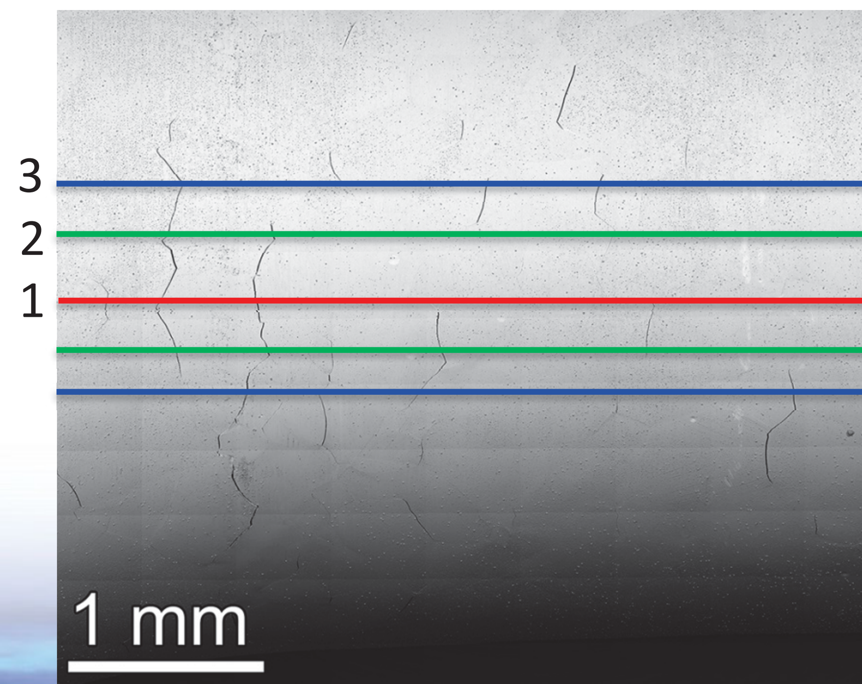
Observation of Surface and Cross Sections to Determine Crack Morphologies

A. *Gauge length and width amenable to complete examination.*
B. *Highly polished condition enabling detailed surface exams.*
C. *Specimen intermittently removed and crack surface lengths measured.*
D. *After DCPD detection of initiation, specimens are sectioned to intersect desired cracks.*
E. *Serial polishing (50-200 µm steps) performed to observe crack depth, shape, and path.*
F. *More than 500 crack observations for some specimens.*





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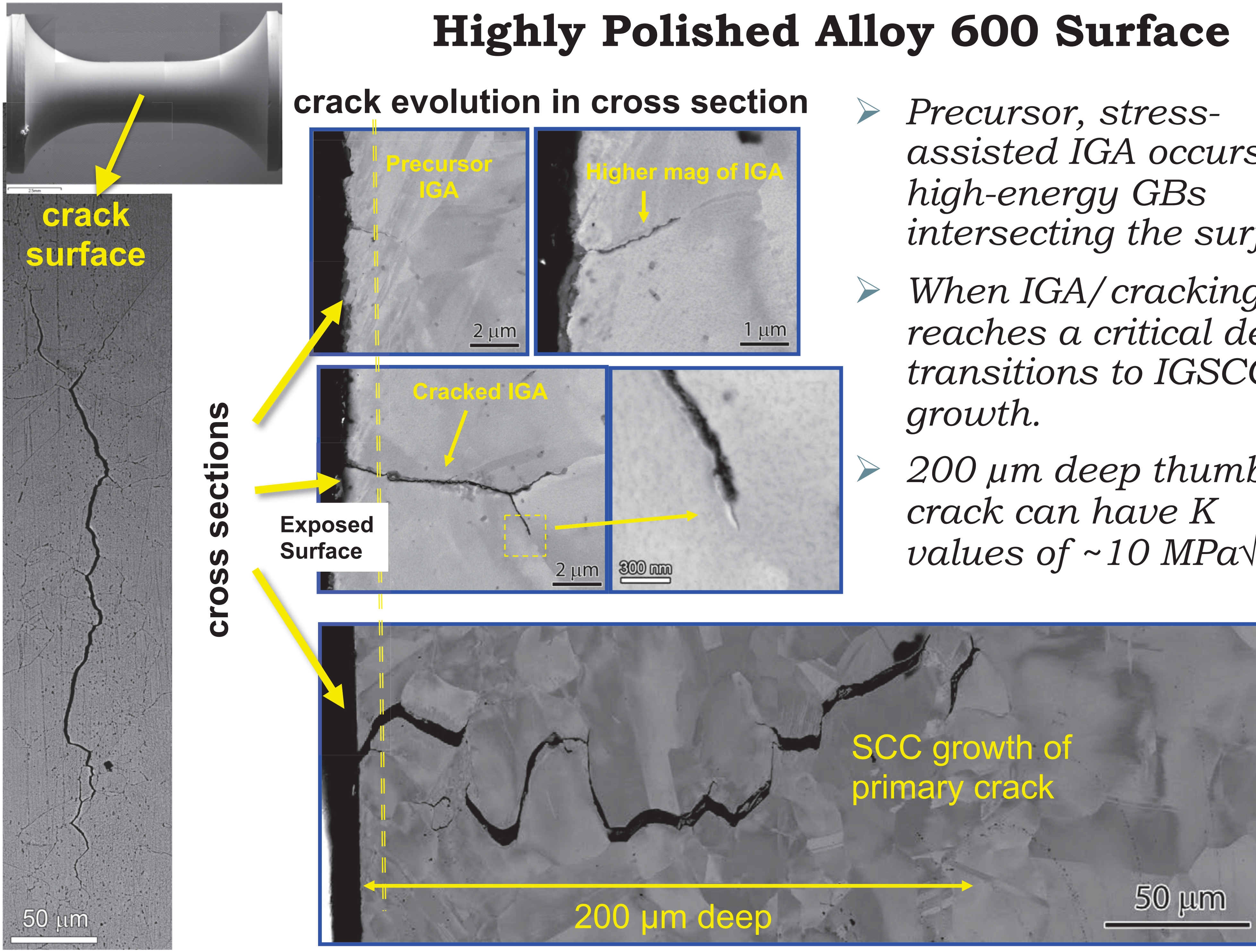
Evolution of SCC Crack Initiation from Highly Polished Alloy 600 Surface

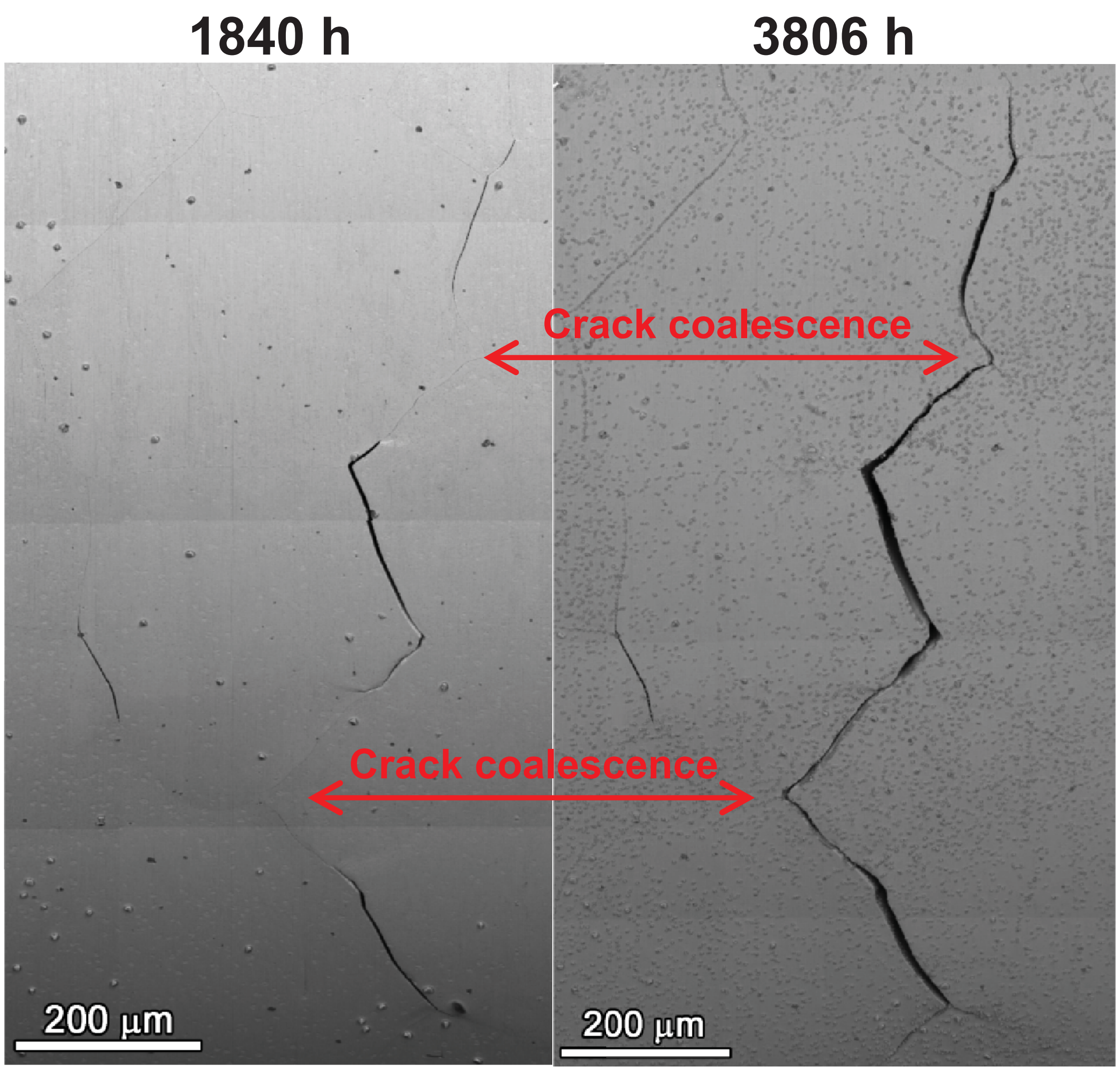


- *Precursor, stress-assisted IGA occurs at high-energy GBs intersecting the surface.*
- *When IGA/cracking reaches a critical depth, transitions to IGSCC growth.*
- *200 µm deep thumbnail crack can have K values of ~10 MPa√m.*

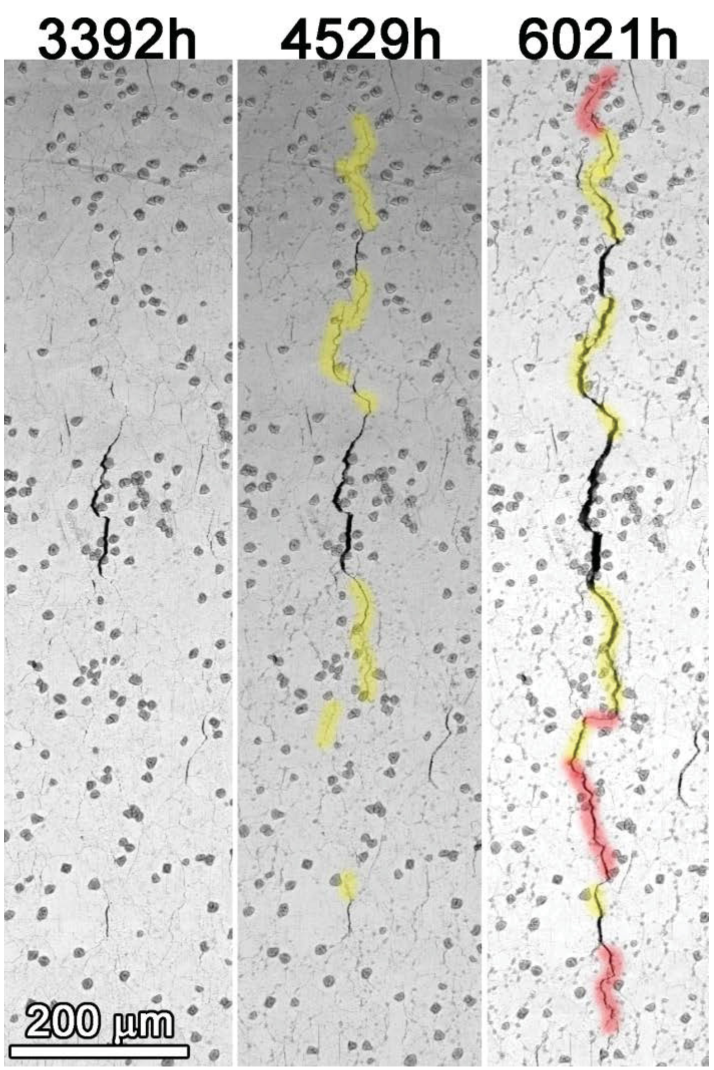
Crack Coalescence on the Surface

- *Multiple sites of crack growth and coalescence in non-cold worked alloy 600.*

IN048 (AR Alloy 600 CRDM)



IN013 (AR Alloy 600 Plate)



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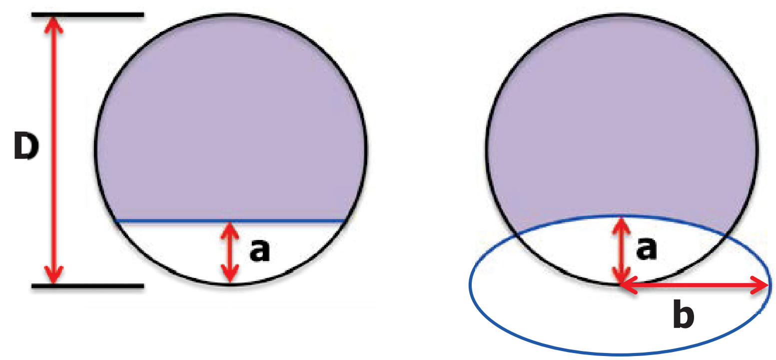
- *Low K SCC crack growth rate testing*

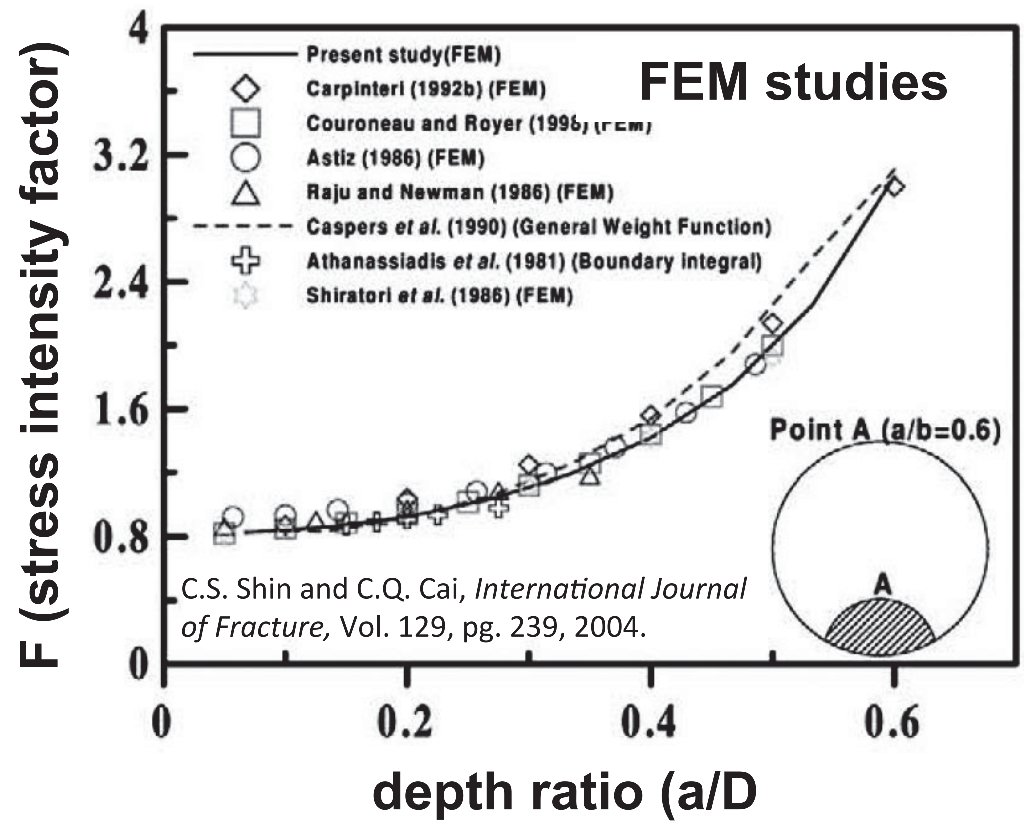
Stress Intensity Estimates and Relationship to Initiation Response

- *Ongoing effort to perform detailed measurements of crack morphology.*
- *Crack shape information being used to estimate stress intensity at time of initiation.*
- *Early measurements indicating a critical stress intensity of ~10 MPa√m for transition to higher indicated DCPD strain rate response.*
- *Consistent with French experience of ~9 MPa√m for transition to higher SCC crack growth susceptibility.*

straight crack | elliptical or circular crack



$$K = \underline{F}\sigma\sqrt{\pi a}$$



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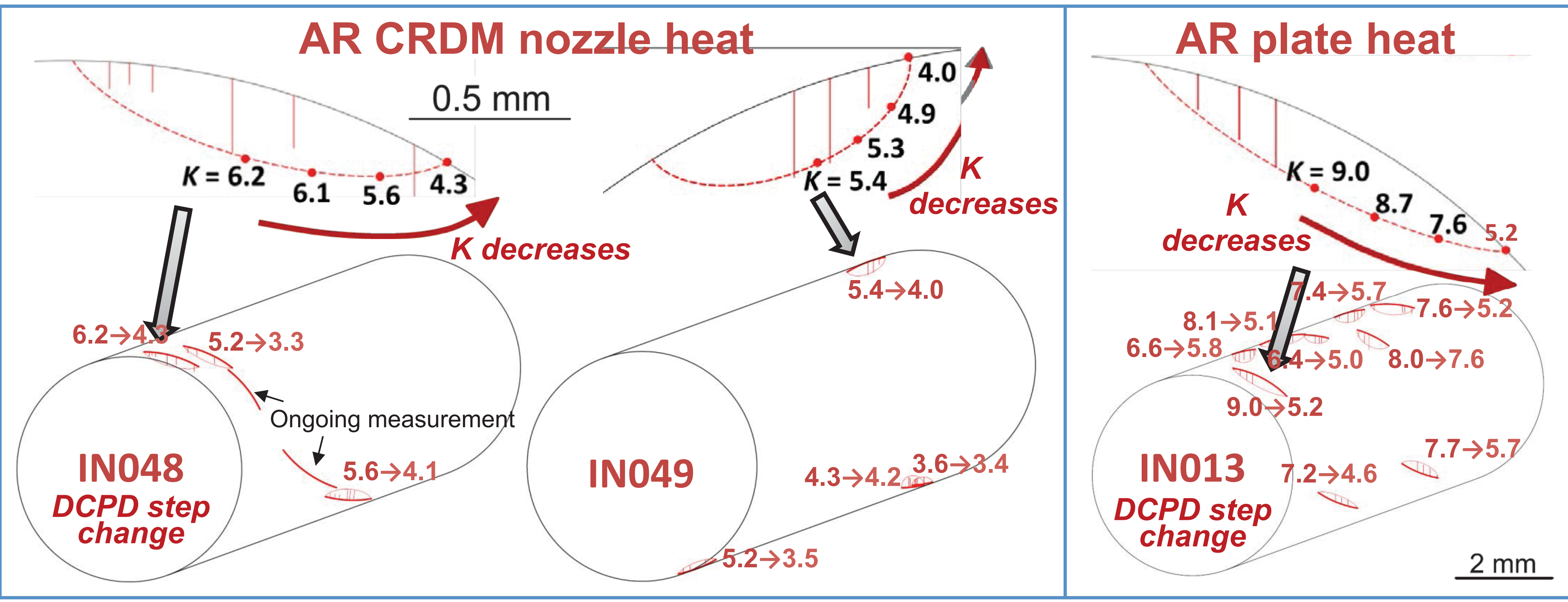
Preliminary Estimation of Local K at Crack Fronts in non-CW Specimens

- ***K determined from finite element modeling (FEM)*** *[Shin 2004, IJF 129]*
  - *K values normally decrease from crack center to crack surface.*
  - *Lower K levels in non-CW specimens are consistent with limited SCC growth and shallow cracks during initiation tests.*

Schematics illustrating location and depth profiles for several large circumferential cracks on gauge surface of the three MA alloy 600 specimens.



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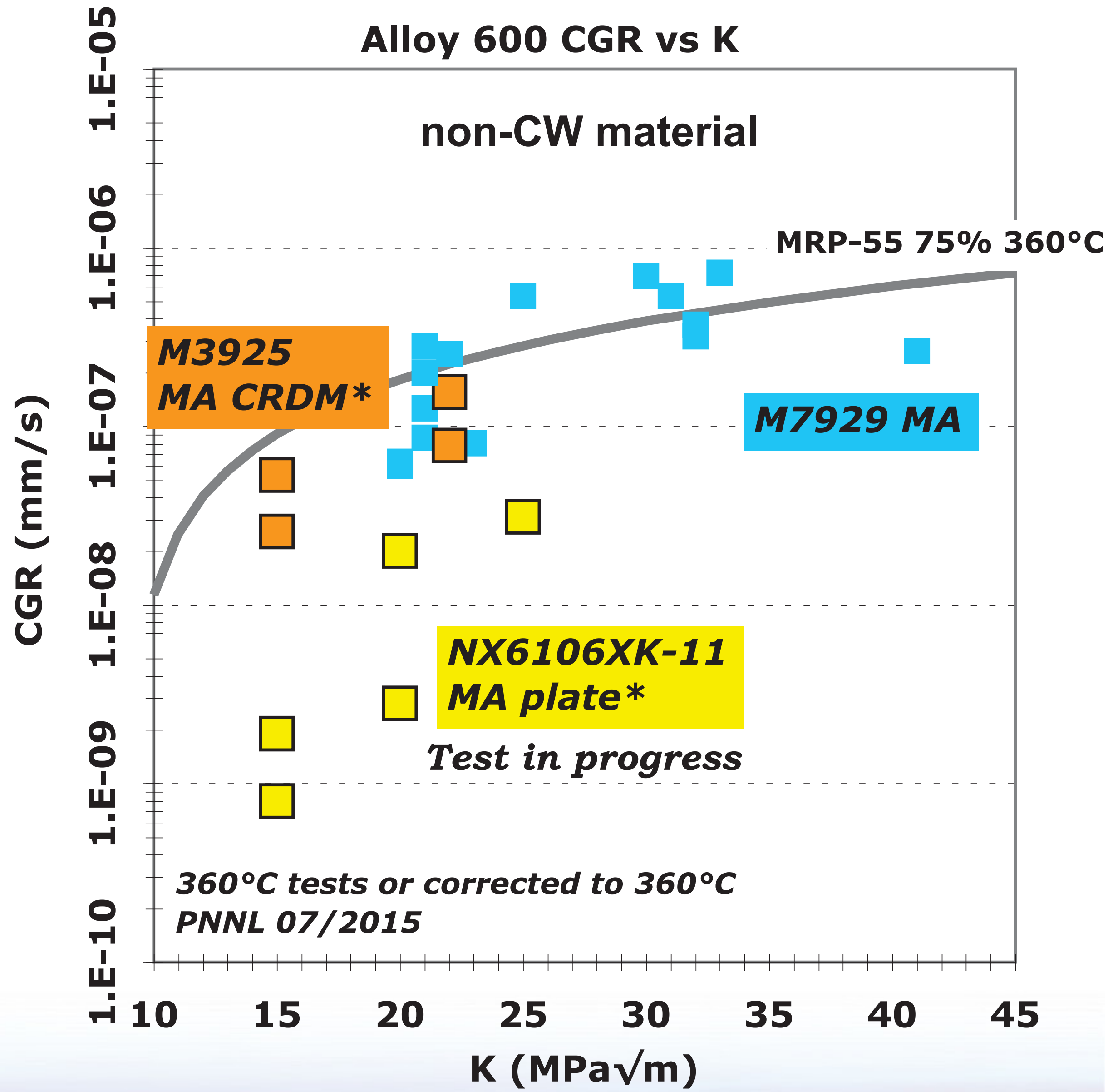
- ***Low K SCC crack growth rate testing***

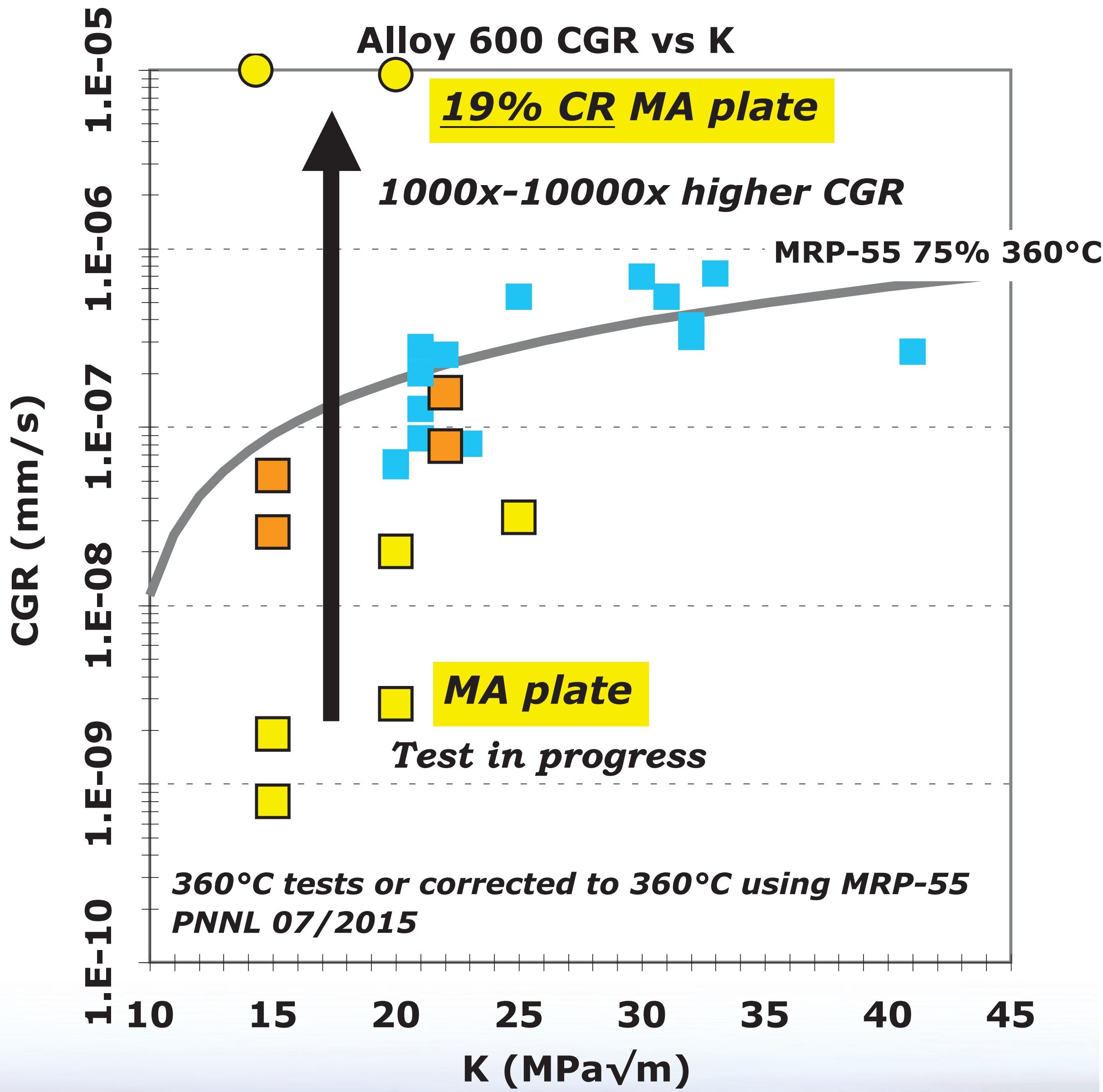
Low K SCC CGR Testing of non-CW Material

- *Low K SCC CGR response being established using compact tension specimens.*
- *Compare to crack initiation times and initiation behavior.*
- *Several materials are the same as used for initiation studies.*
- ***SCC CGRs of non-CW materials decrease significantly as K level drops below ~20 MPa√m.***



Effect of Cold Work on Low K SCC Crack Growth Rates

- *Effect of CW: Preliminary results indicate ≥1000x higher CGR for 19% CR material.*
- *Tests continuing to better establish low K response.*
- ***Difference between CW and non-CW CGR is consistent with strong difference in initiation time between CW and non-CW.***



Summary

- *A significant reduction in SCC initiation time was observed for ~7-8% CW alloy 600 loaded to its YS.*
  - *Need to understand relative contributions from (1) applied stress and (2) effect of CW on material susceptibility.*
- *Low K SCC CGR testing in progress. Current observations show that at low K, CW materials exhibit ≥1000x CGR than non-CW material.*
  - *Consistent with strong difference in initiation response between CW and non-CW materials.*
- *In CW materials, IG cracks estimated to have a depth of ~200 μm at the time of initiation with K = ~10 MPa√m.*
  - *Work is ongoing but suggests that DCPD-based initiation response is tied to a critical stress intensity for accelerated SCC crack growth.*

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SCC Initiation Research on Cold-Worked Alloy 690: Grain Boundary Creep Cavities and IGSCC Precursors

Light Water Reactor Sustainability

Ziqing Zhai, Matt Olszta, Dan Schreiber, Karen Kruska, Mychailo Toloczko and Steve Bruemmer

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Light Water Reactor Sustainability R&D Program

Recent Alloy 690 Activities and Highlights

- *High-Temperature Autoclave Test Systems*
  - *Three dedicated SCC initiation systems for LWRS operational including one running 36 actively loaded tensile specimens. Two smaller systems being upgraded to test 6 actively loaded tensile specimens.*
- *Corrosion Experiments – Precursor Damage*
  - *Alloy 690: completed examinations of protective Cr_2O_3 film formation above high energy grain boundaries. No IGA, but extensive grain boundary migration and Cr depletion below surface oxide. Experiments ongoing evaluating IG corrosion response if surface oxide is removed by FIB.*
- ***SCC Initiation Experiments***
  - ***Alloy 690:*** *blunt-notch and constant load experiments ongoing on CW alloy 690 materials susceptible to SCC growth. IG creep cavitation during dynamic straining promotes SCC initiation for blunt notch. Evidence for grain boundary cavities and IGSCC precursor damage during long-term, constant-load tests.*

Definitions of Crack Initiation (Andresen/Scott)

Scientific Definition	Formation of a mechanical distinct geometry that will tend to grow in preference to its surroundings as a sharp crack.
Practical Definition	Detectability that is likely to be achieved in-situ for laboratory autoclave experiment
Phenomenology - Environment	Chemically short cracks are cracks in which a mature, long crack chemistry has not formed.
Phenomenology - Mechanics	Mechanically small cracks are surface cracks growing under plane stress conditions, or where linear elastic fracture mechanics does not directly apply.
Phenomenology - Metallurgy	Metallurgically small cracks are cracks smaller than one grain diameter or other metallurgical features of importance.

Proposed Definition (by Andresen/Scott): Formation of a mechanically distinct geometry that will tend to grow in preference to its surroundings.

Critical Issue: SCC initiation occurs when cracks reach a size/depth where the stress intensity is sufficient for stable and significant SCC growth.

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Outline

- Blunt-Notch SCC Initiation Tests
  - Phase 1: 31%CF Alloy 690 CRDM Heats
  - Phase 2: 31%CF and 21%CF Alloy 690 CRDM Heats
  - Characterization of IG Crack Nucleation & Growth
- Tensile Constant Load SCC Initiation Tests
  - Long-Term Exposures of 12%CF, 20%CF and 30%CF Alloy 690 CRDM and Plate Heats
  - Surface and Near Surface Characterization of IG Damage Evolution after Test Time of ~1 year

Blunt Notch Materials

- *Specimens were machined from two heats of Alloy 690 CRDM tubing cold forged to different degrees*

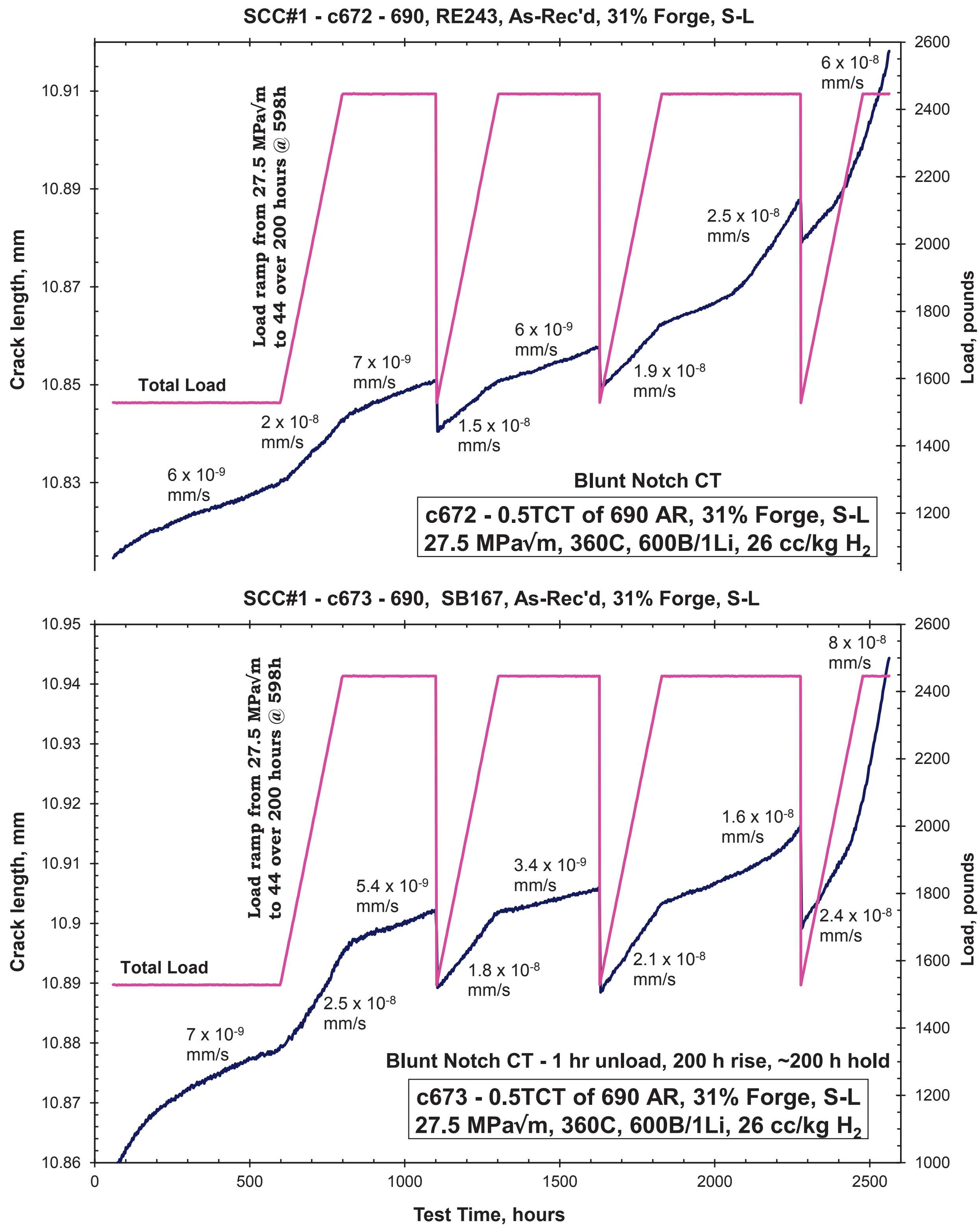
Material and Heat Number	Description	Composition, wt%	Heat Treatments and Cold Work Level
Valinox Alloy 690TT RE243	CRDM Tube 2360 1.4" wall	Ni-28.9Cr-10.4Fe-0.02C-0.3Mn-0.35Si-0.14Al-0.23Ti-0.024N-0.008P-0.0005S	1122°C/~1 min, WQ + 716-725°C/10.5 h, air cool 12.7%, 21%, 31%CF
Sumitomo Alloy 690TT E67074C	CRDM Tube 1.31" wall	Ni-29.8Cr-9.8Fe-0.020C-0.29Mn-0.23Si-0.0002S	725°C/10 h/AC 21%, 31%CF

- *As-received TT microstructure*

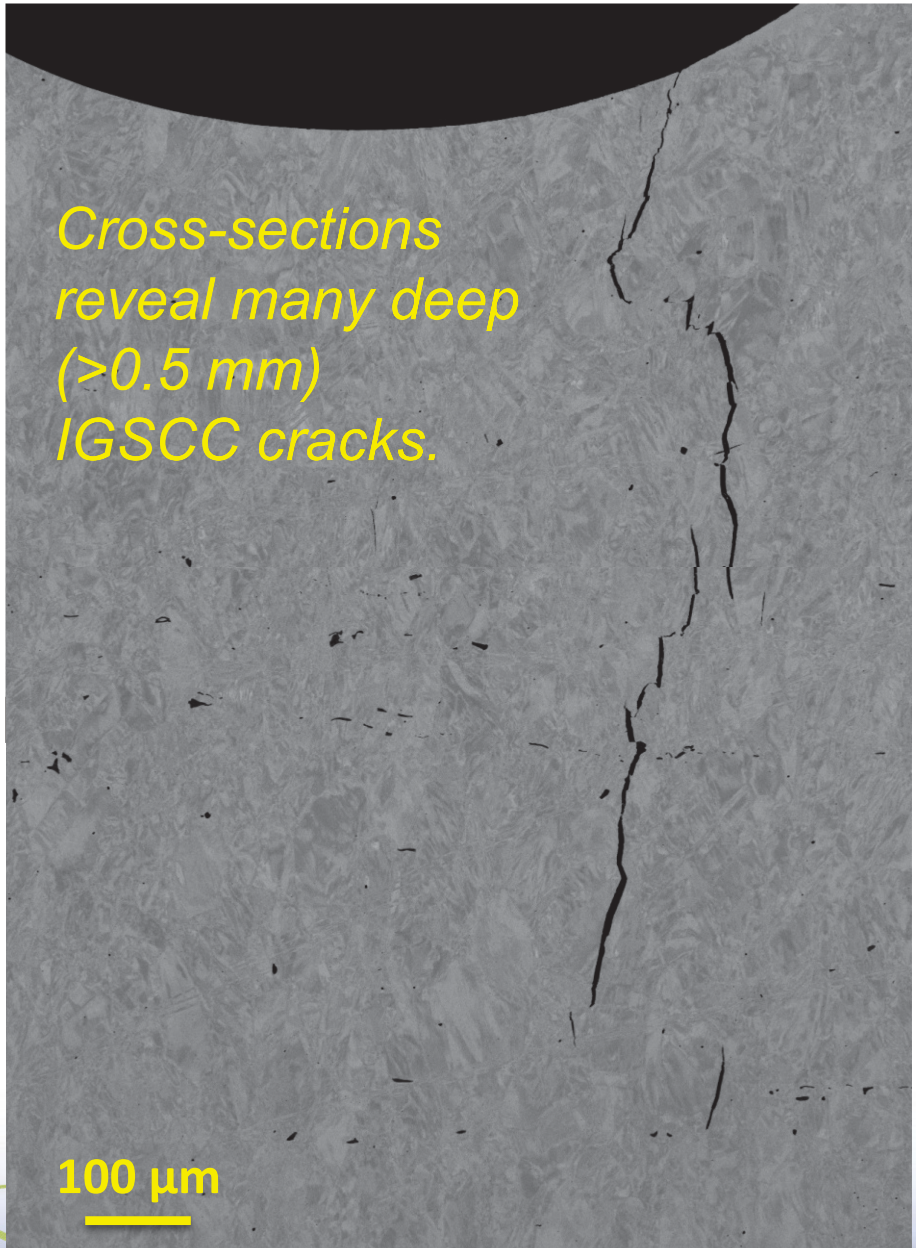
Sample	Microstructure
Valinox CRDM Tube Alloy 690TT RE243	Equiaxed, large grains (~90 µm in diameter); semi-continuous (discrete + cellular) IG carbides and Cr depletion to ~21 wt%, few IG and matrix TiN
Sumitomo CRDM Tube Alloy 690TT	Equiaxed, smaller grains (~35 µm in diameter) with twins; Semi-continuous (discrete + occasional cellular) IG carbides; isolated matrix TiN

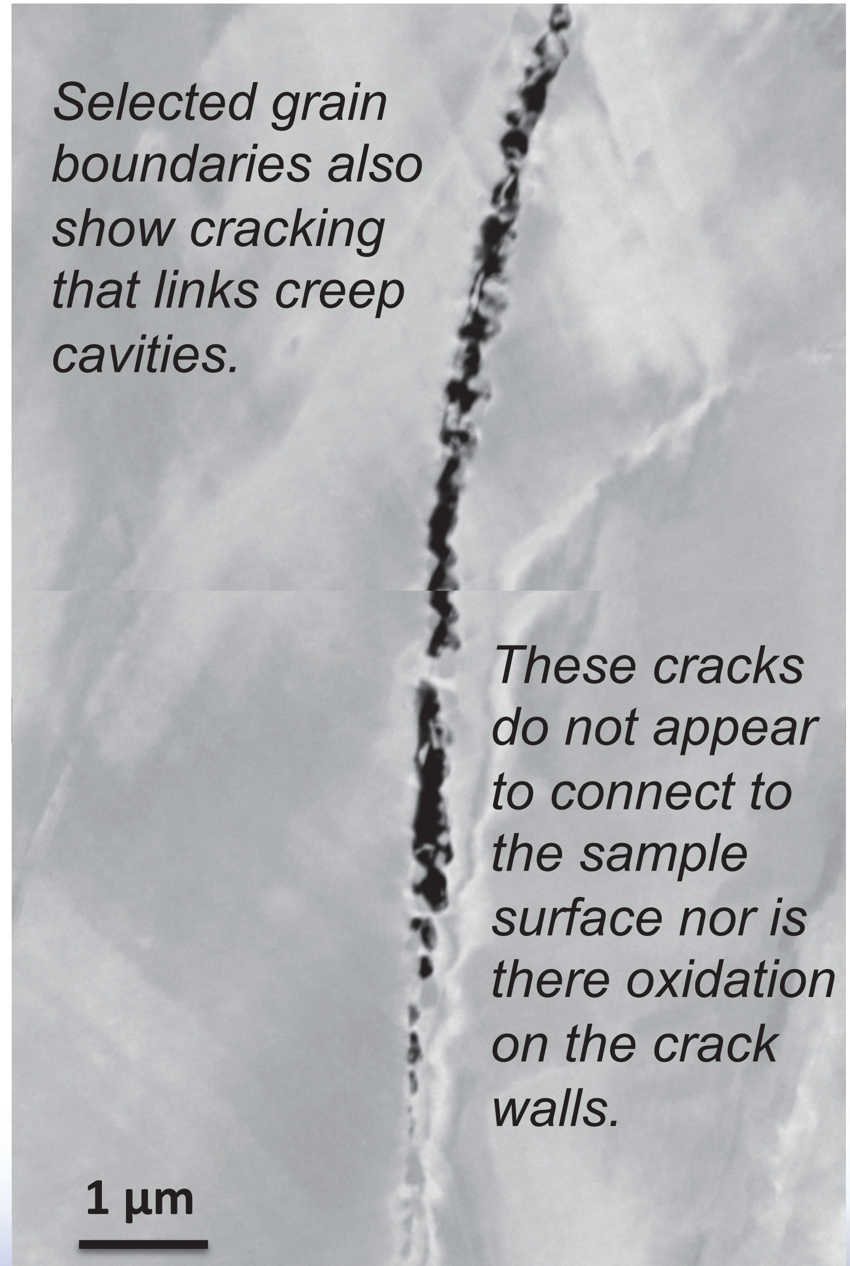
Phase 1 Blunt-Notch Tests at GEG R&D

- *Peter Andresen is conducting several blunt-notch initiation tests under LWRS funding.*
- *Same PNNL CW alloy 690 materials.*
- *Phase 1: 27.5-44 MPa√m ramp over 200 hours followed by varying hold.*
- *FEM estimated ~1.0% plastic strain during ramp.*
- *Clear **crack initiation and growth** in the 3rd hold period.*
- *Extensive IG cracking to depths >0.5 mm.*



Crack Identification in Cross-Sections from 31%CF Alloy 690 Blunt-Notch Specimen





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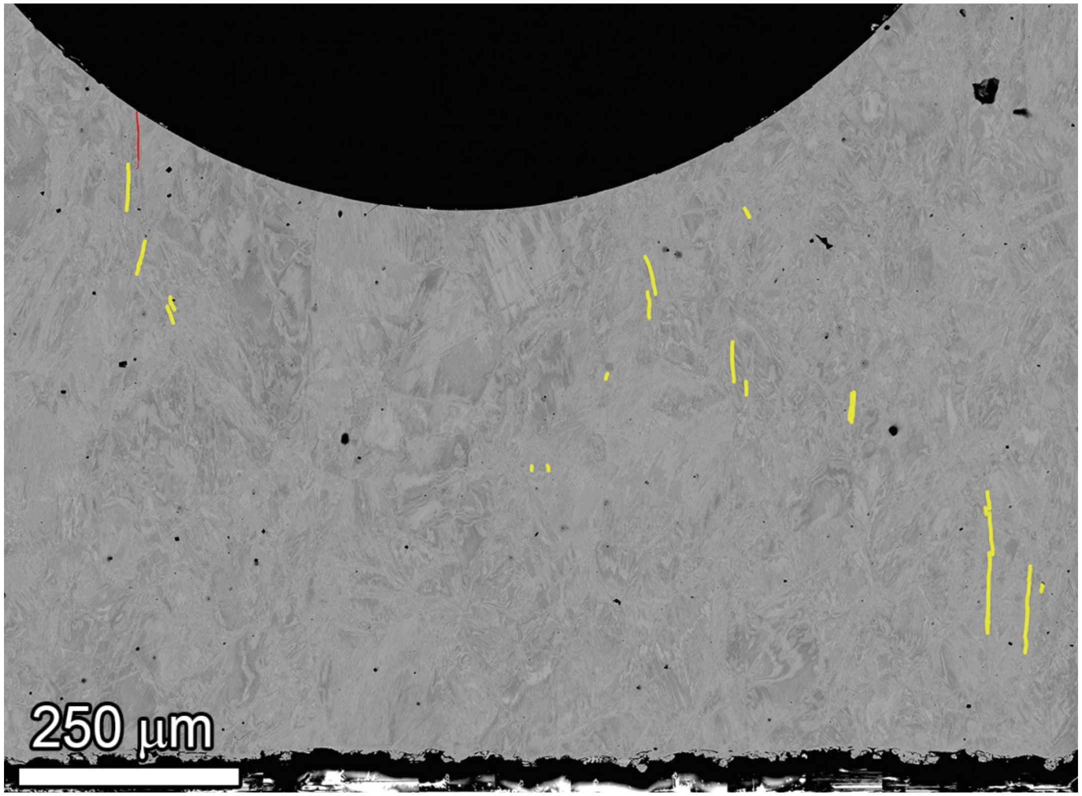
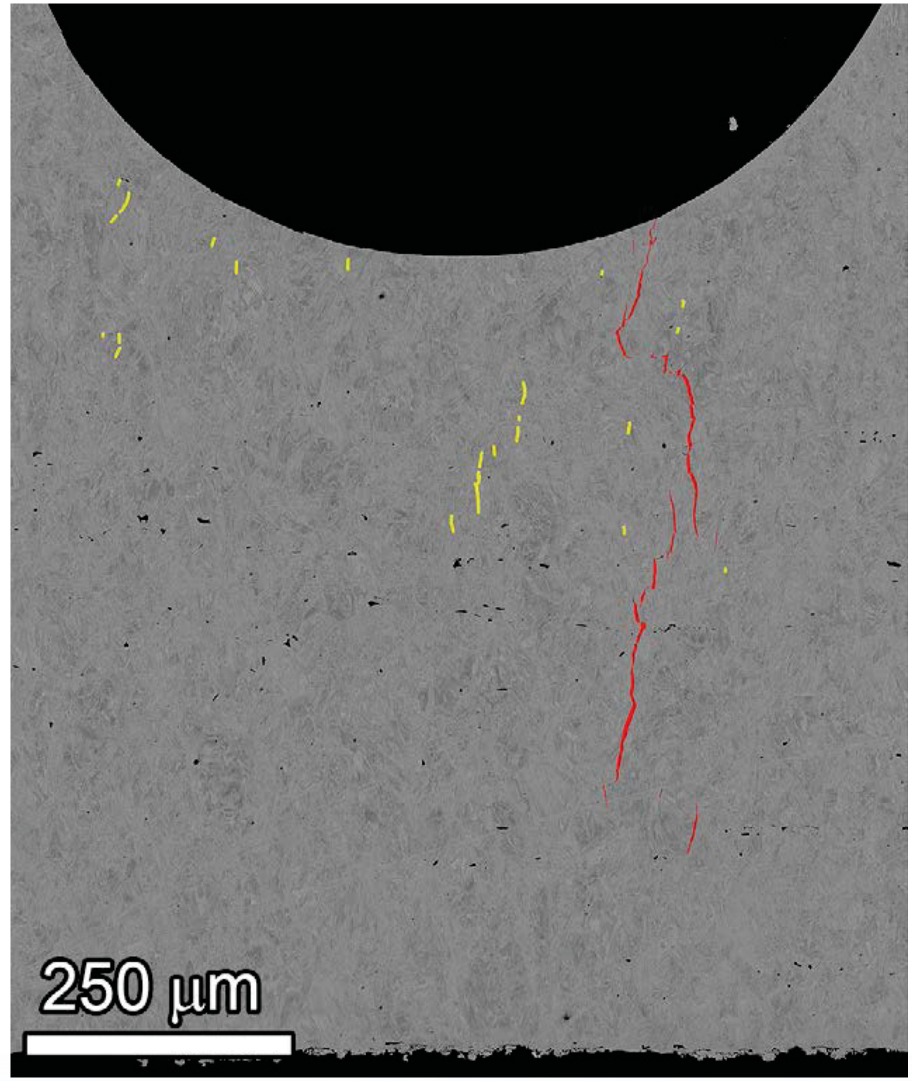


Slice #2

*Cracks identified as creep (**yellow**) and SCC (**red**) are highlighted.*



Slice #4

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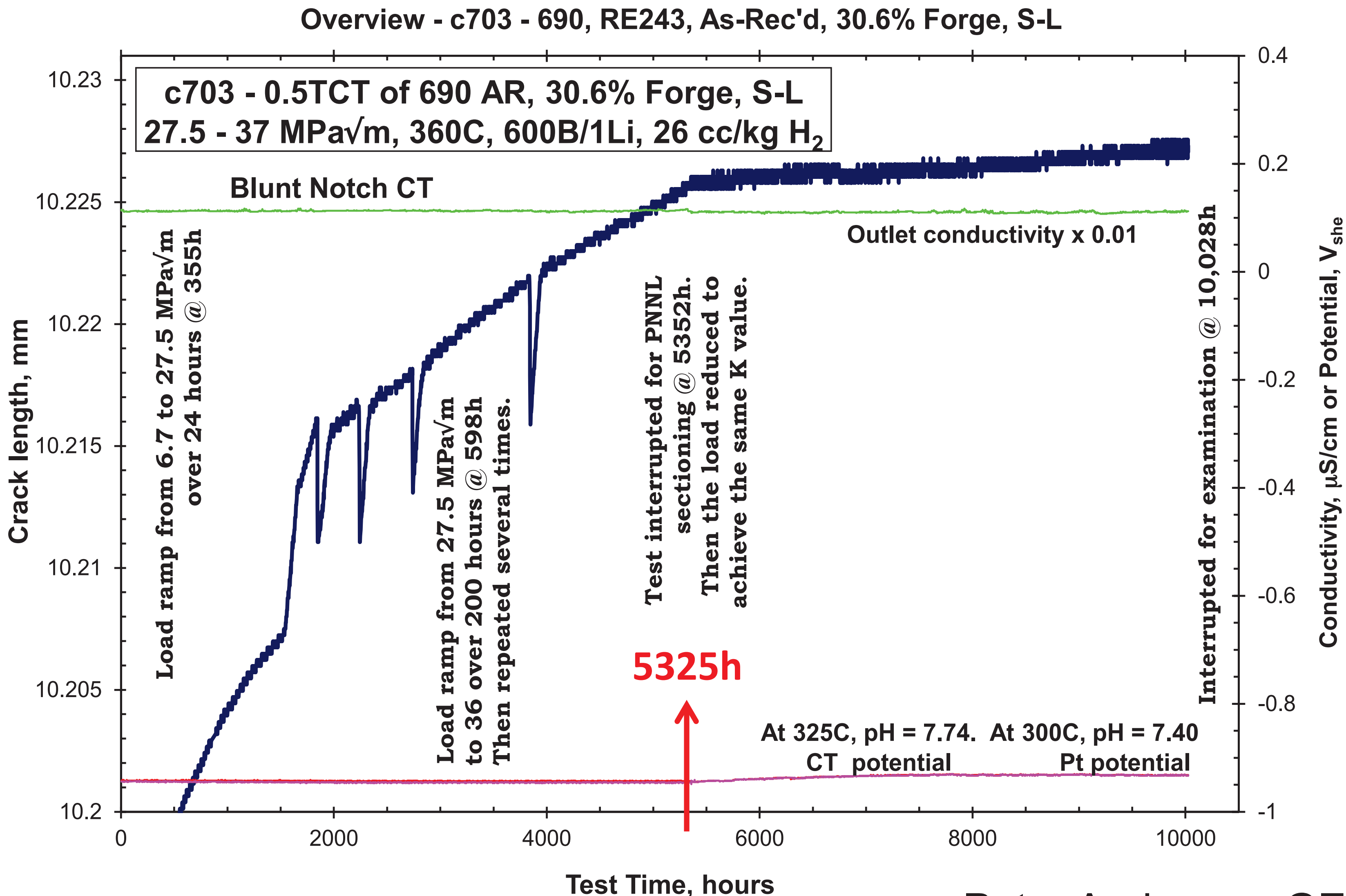
Phase 2 Blunt-Notch Tests at GEG R&D

- 0 - 5,325 h: 4-6 loading ramps at K = 27.5 to 36 MPa√m + ~500 h hold
- 5,325 - 10,000 h: Constant load at K = 37 MPa√m



~0.5% plastic strain during loading ramps based on FEM

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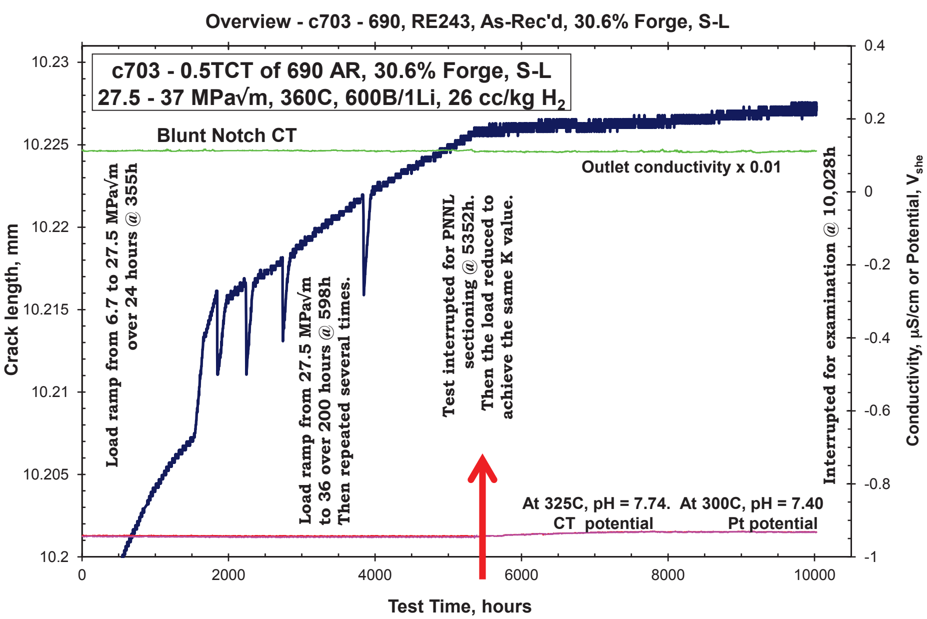
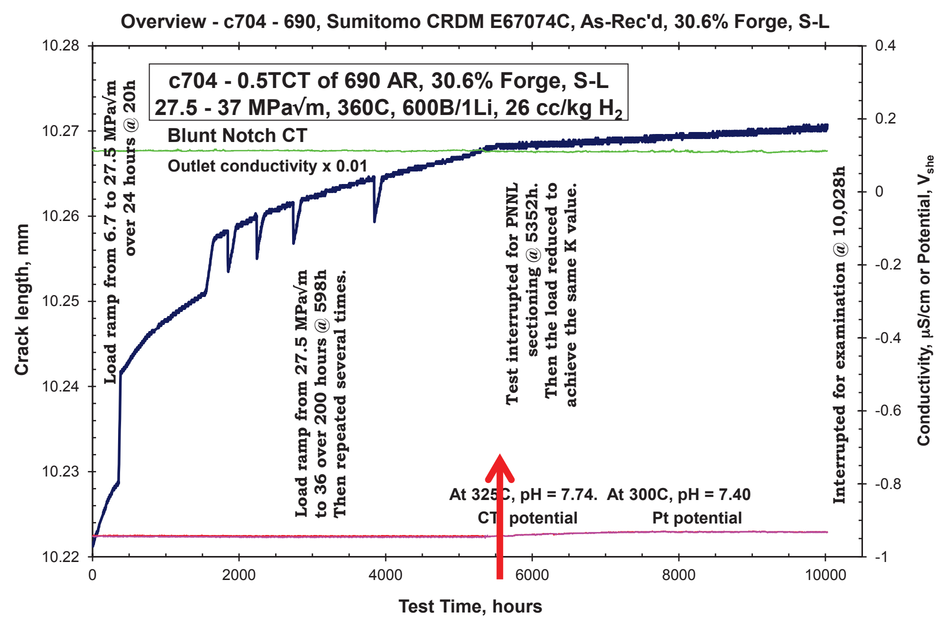
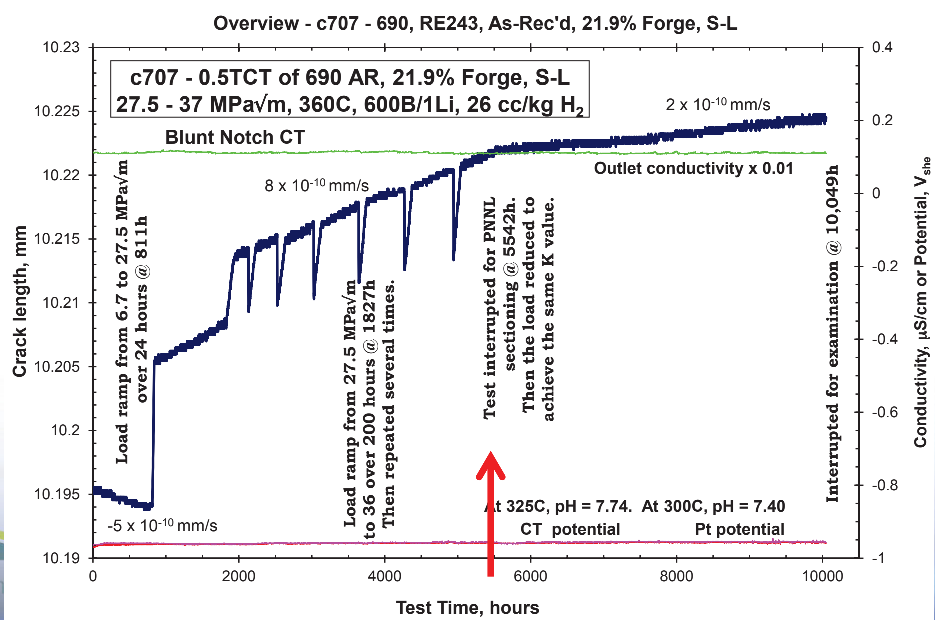
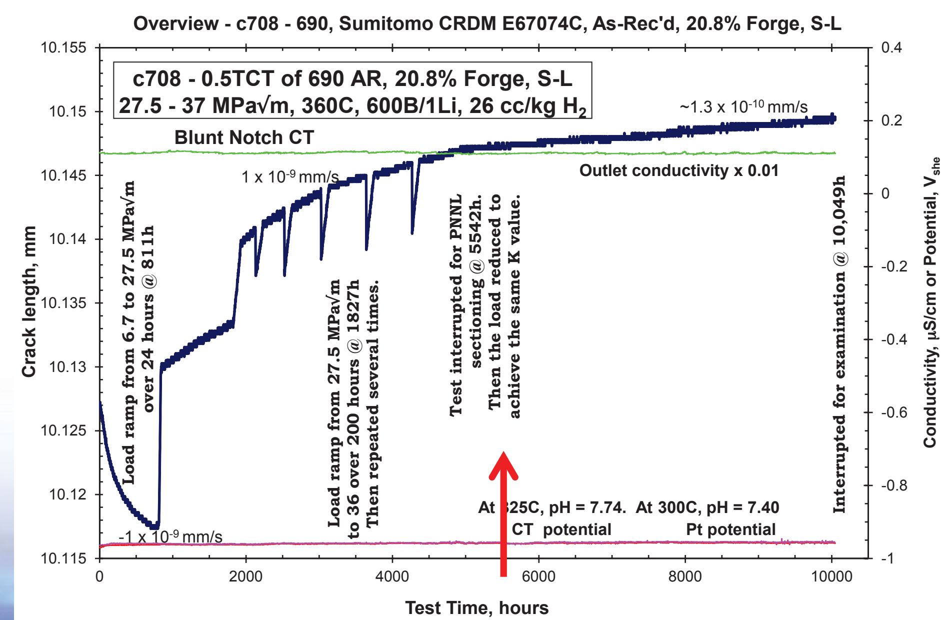
Phase 2 Blunt-Notch Tests at GEG R&D

- 0 - 5,325 h: 4-6 loading ramps at K = 27.5 to 36 MPa√m + ~500 h hold
- 5,325 - 10,000 h: Constant load at K = 37 MPa√m









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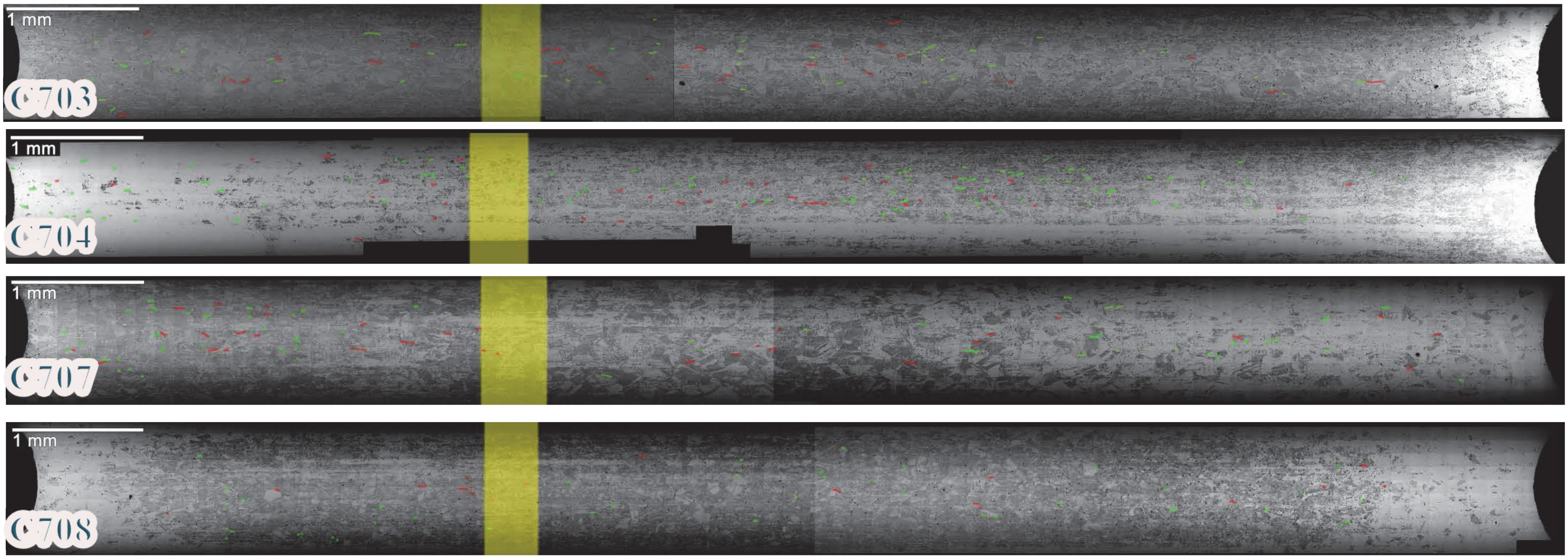
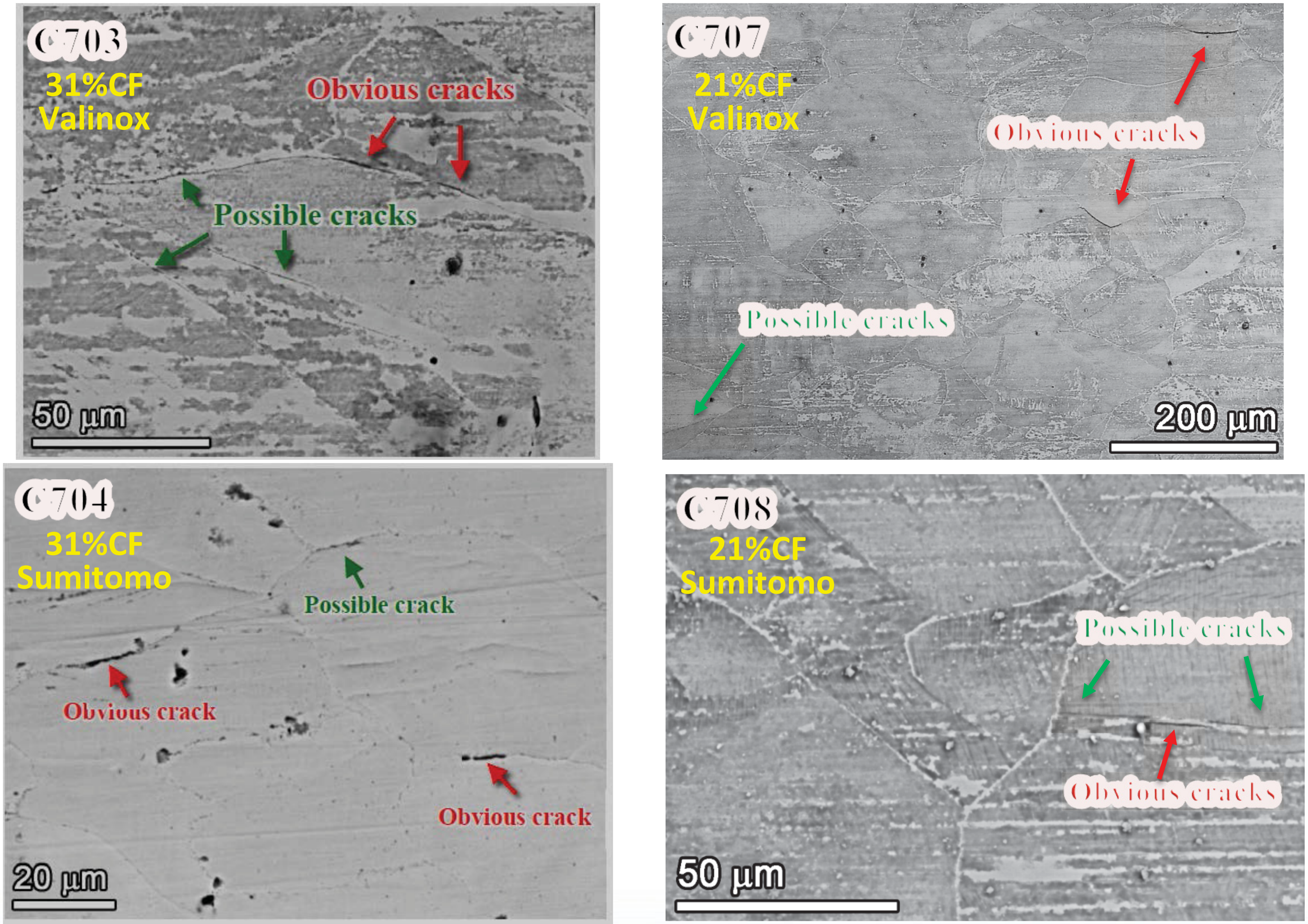
SEM Examinations of Blunt Notch Specimen Surfaces after Exposure Time of 5325 h



1/3rd section removed for serial polishing and SEM exams at PNNL.

2/3rd sections returned to GEG and tests continued at constant K of 37 MPa√m to 10,000 h, ongoing SEM exams.

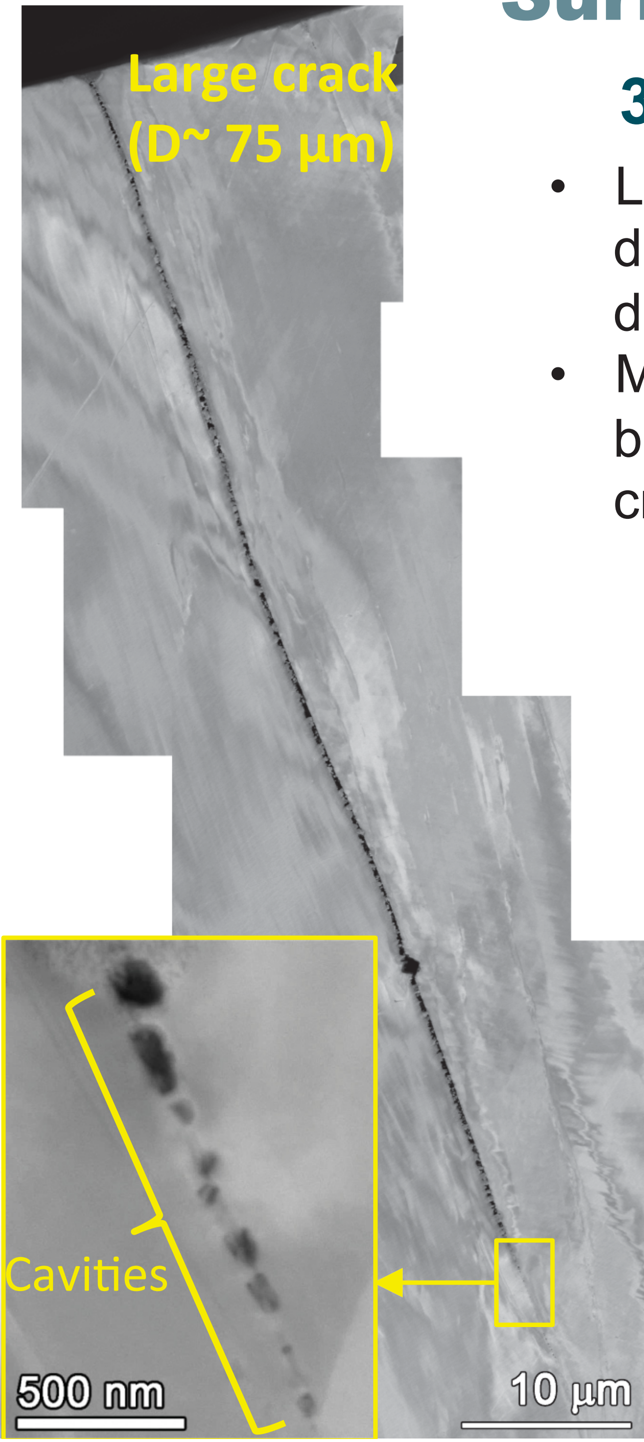
IG Cracks Identified on Blunt Notch Specimen Surfaces after 5325 h Exposure



LWRS Light Water Reactor Sustainability

Low density of IG cracks identified on gauge surfaces for 31%CF specimens, only isolated small cracks for 21%CF specimens.

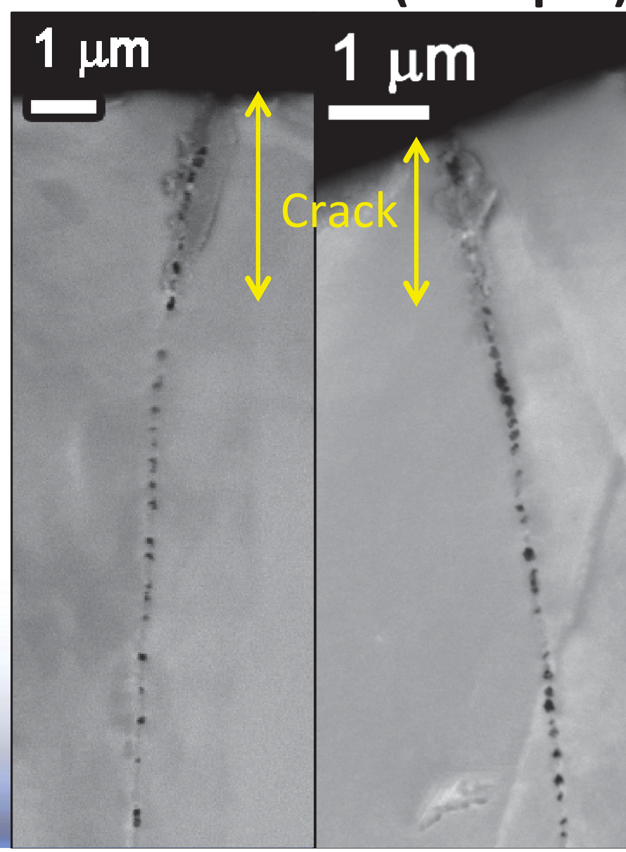
Cross-Section Characterizations of Surface Cracks after 5,325 h



31%CF Valinox

- Low density of larger, >20 µm deep cracks (left), moderate density of small cracks.
- Moderate density of IG cavities below surface and ahead of crack tips.

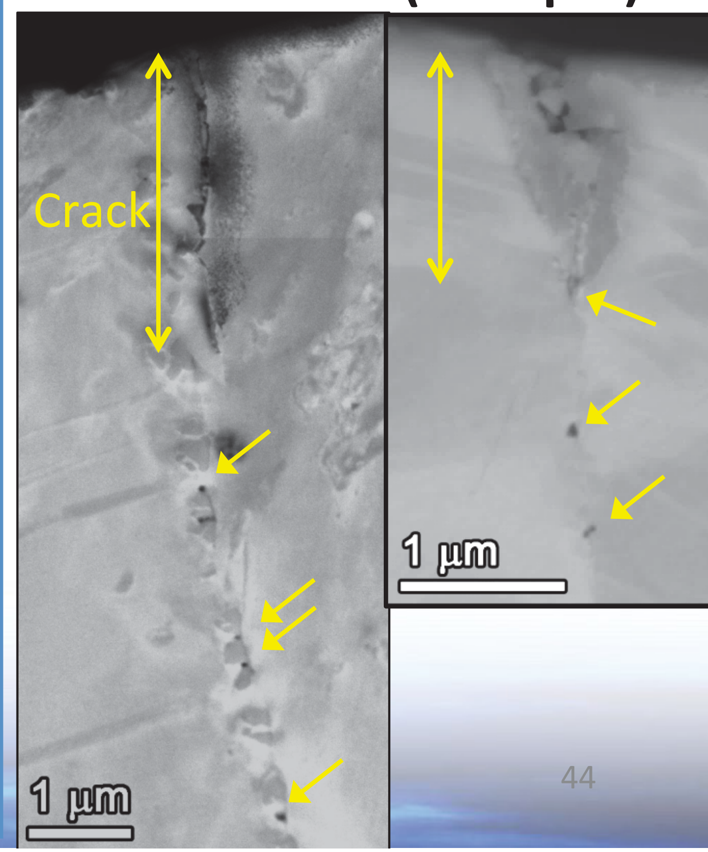
Small cracks (D <3 µm)



21%CF Valinox

- Low density of small, shallow IG cracks.
- Low density of IG cavities below surface and ahead of crack tips.

Small cracks (D <3 µm)



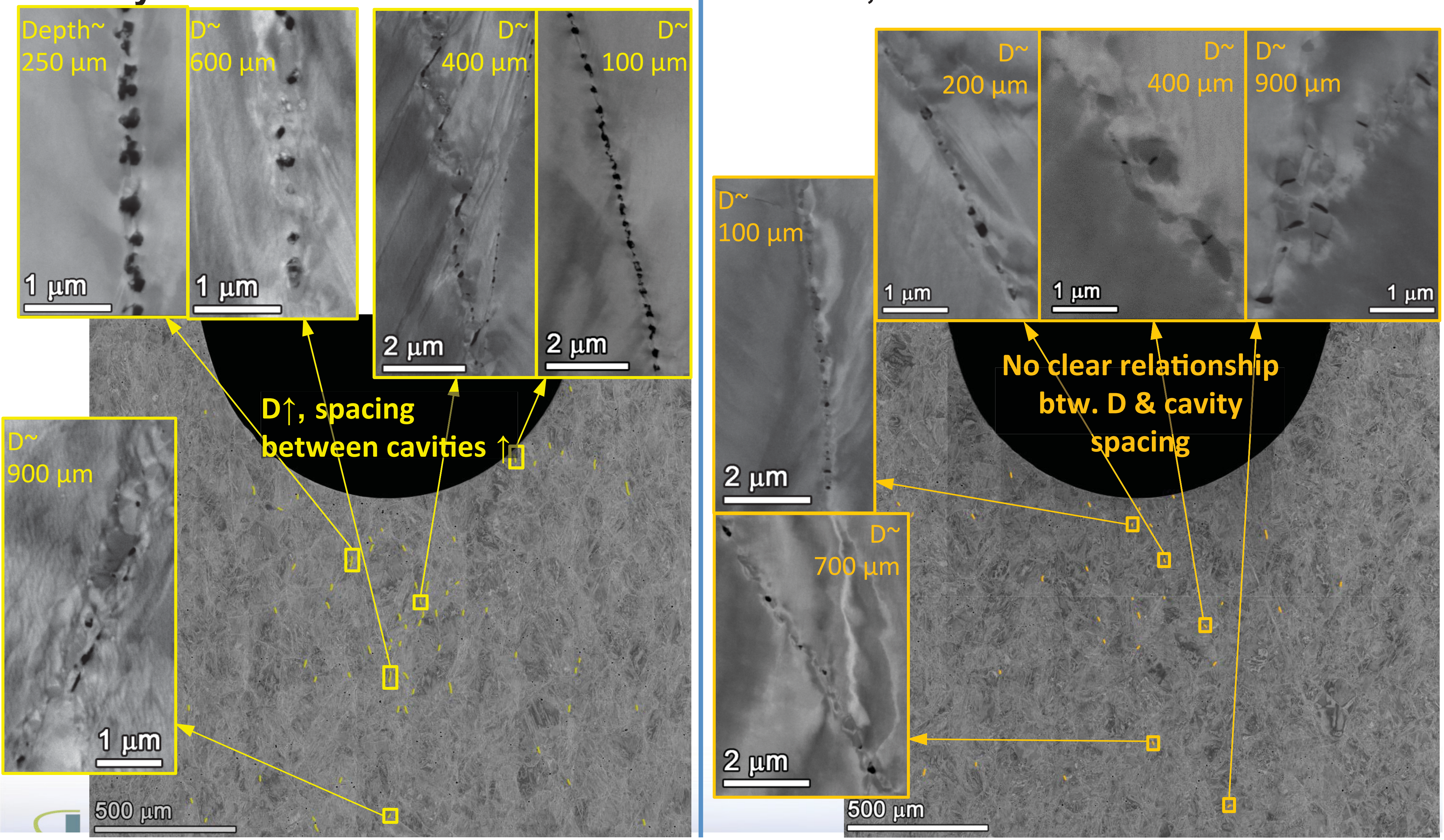
Subsurface Cross-Section Characterizations after 5,325 h

C703 (Valinox 31%CF)

Many semi-continuous IG cavities

C707 (Valinox, 21%CF)

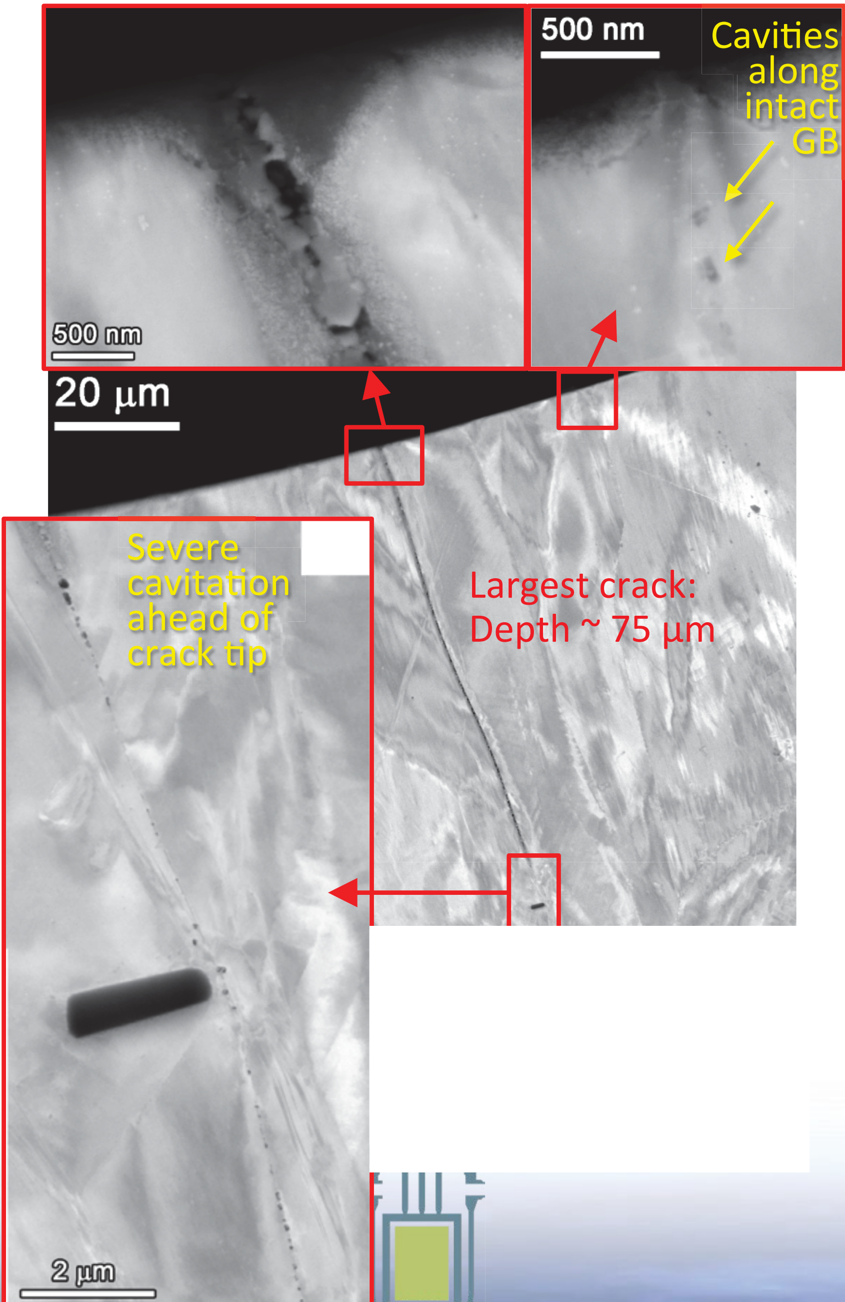
Few, smaller discrete IG cavities

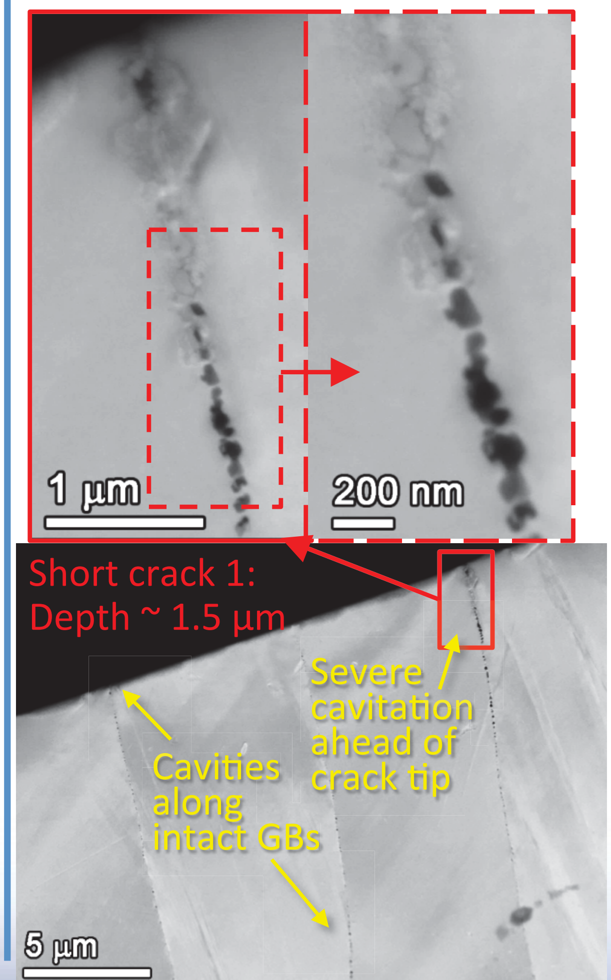


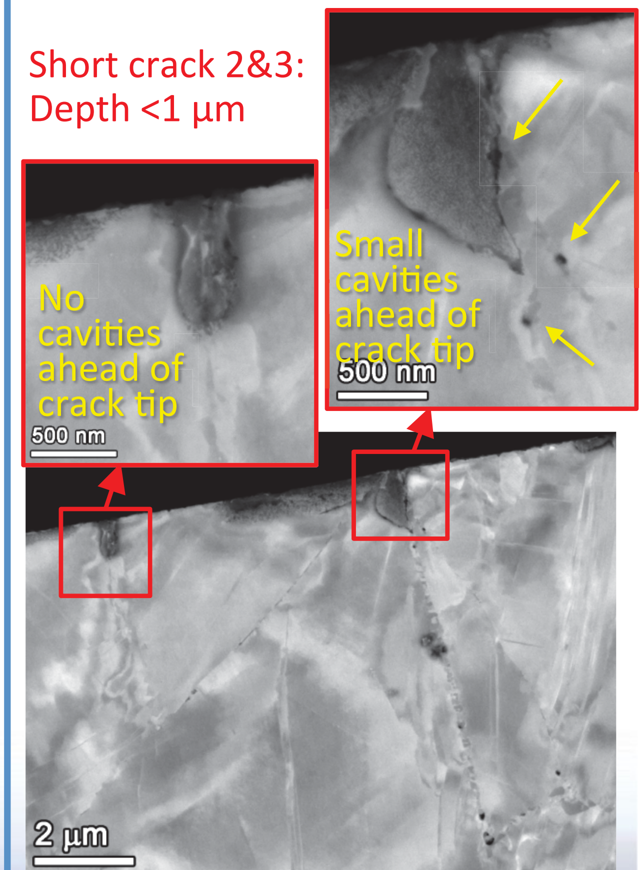
Higher density of creep cavities observed at ~200-500 µm below the surface, consistent with highest stress region in FEM analyses.

Blunt Notch – Typical Crack Morphology on Cross-Section (C703, 31%CF Valinox CRDM)

- Creep cavities were often observed ahead of IG crack tips, especially the larger ones.
- Creep cavities were also found close to the notch surface along some intact grain boundaries.

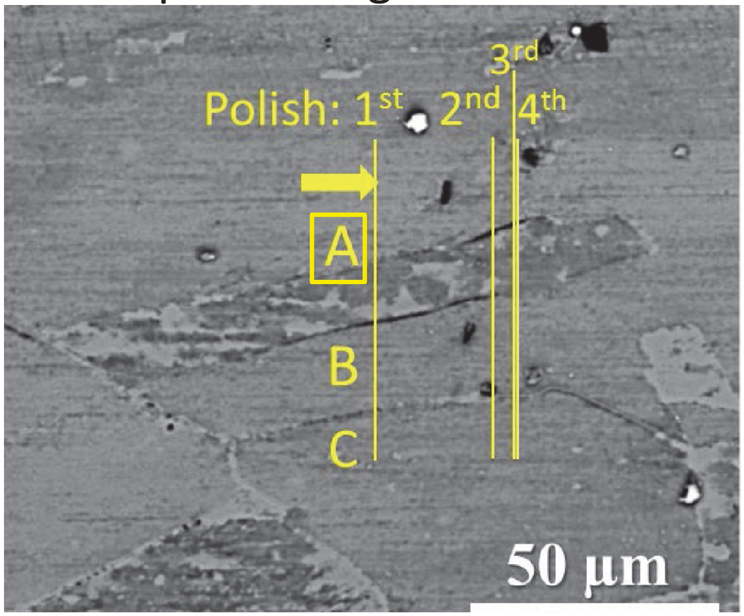


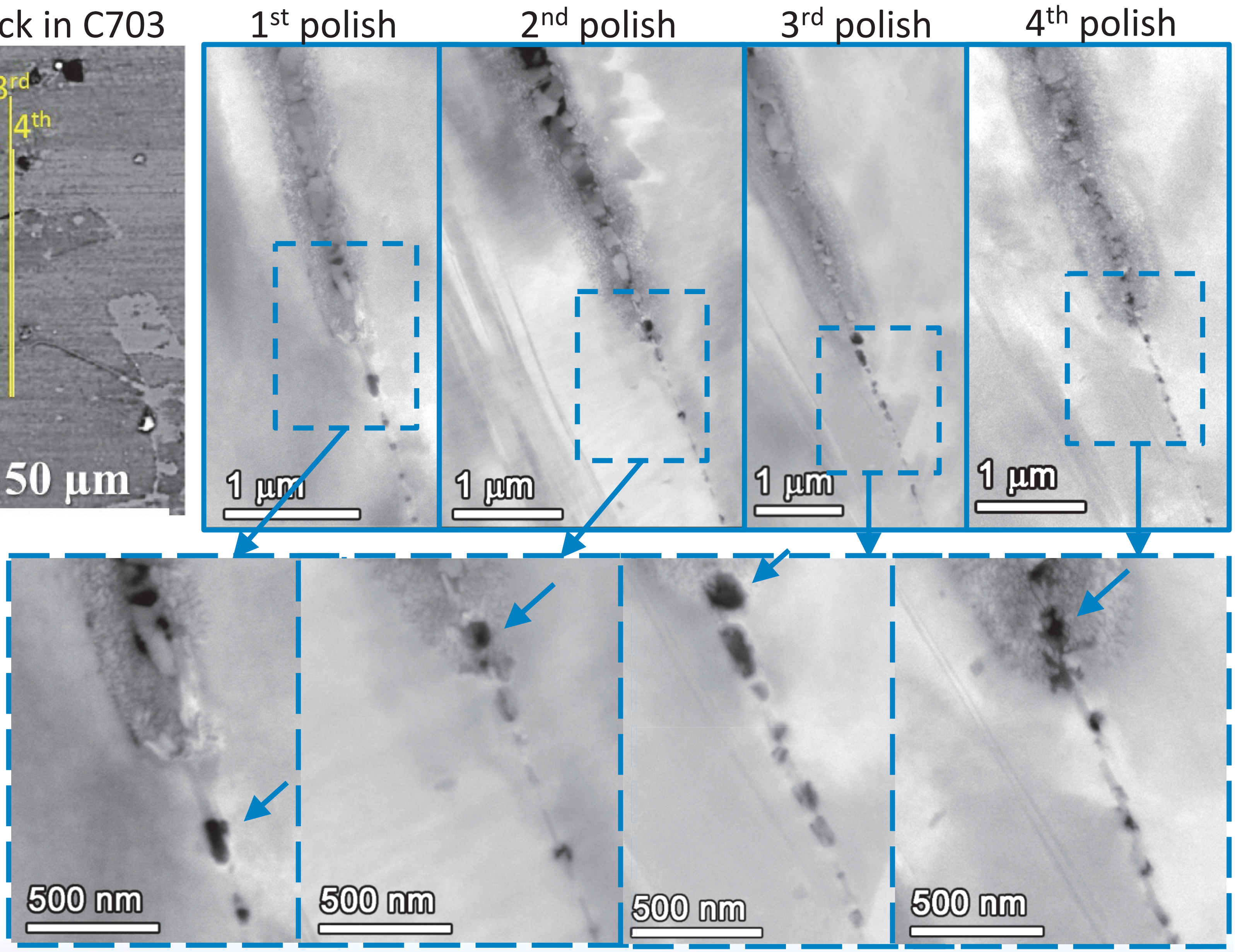




Cross-section Characterizations after 5,325 h

Serial polish: large crack in C703

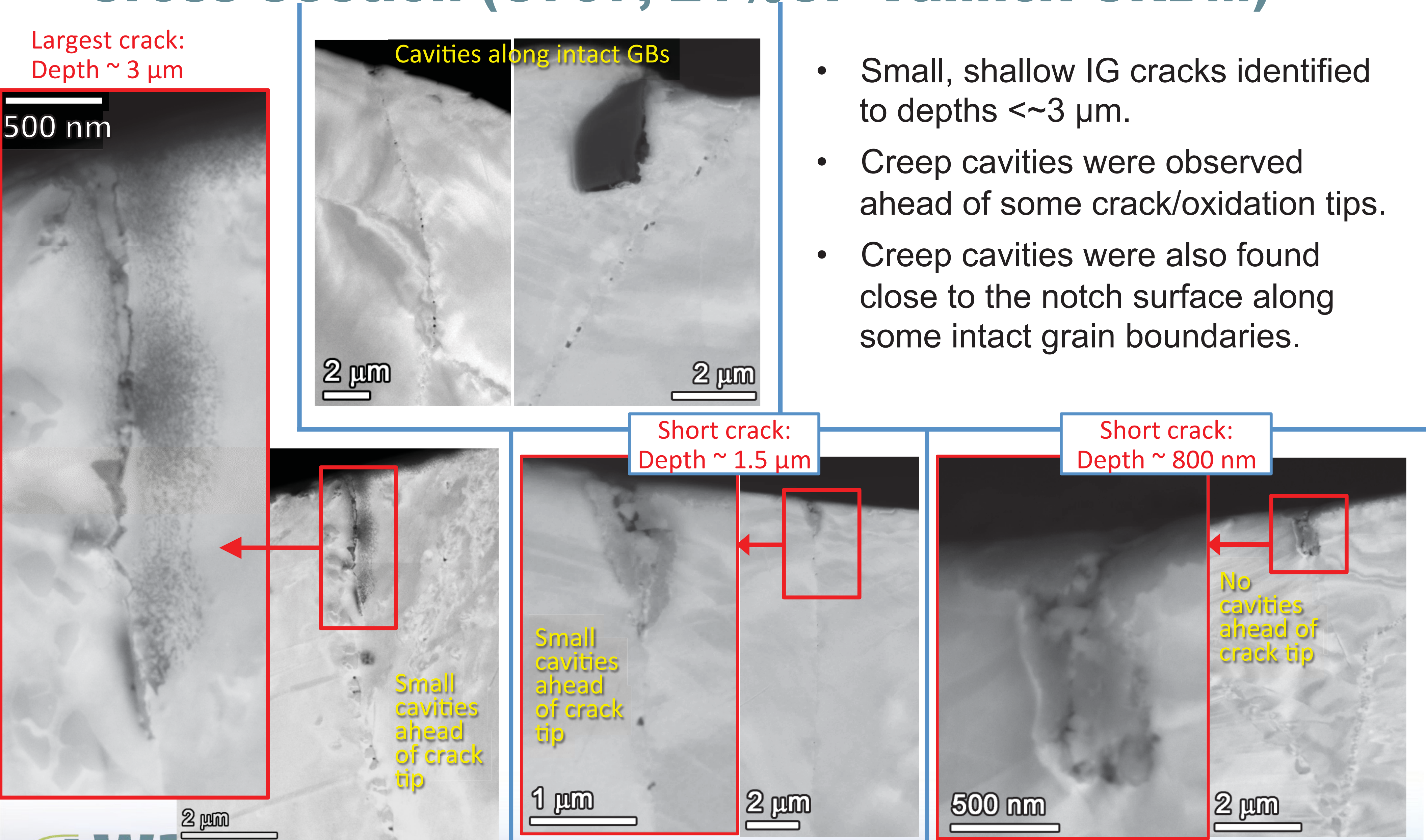




- Largest cavities found closest to the crack/oxidation tip.
- Size and density of cavities drop with distance from crack/ oxidation tip.

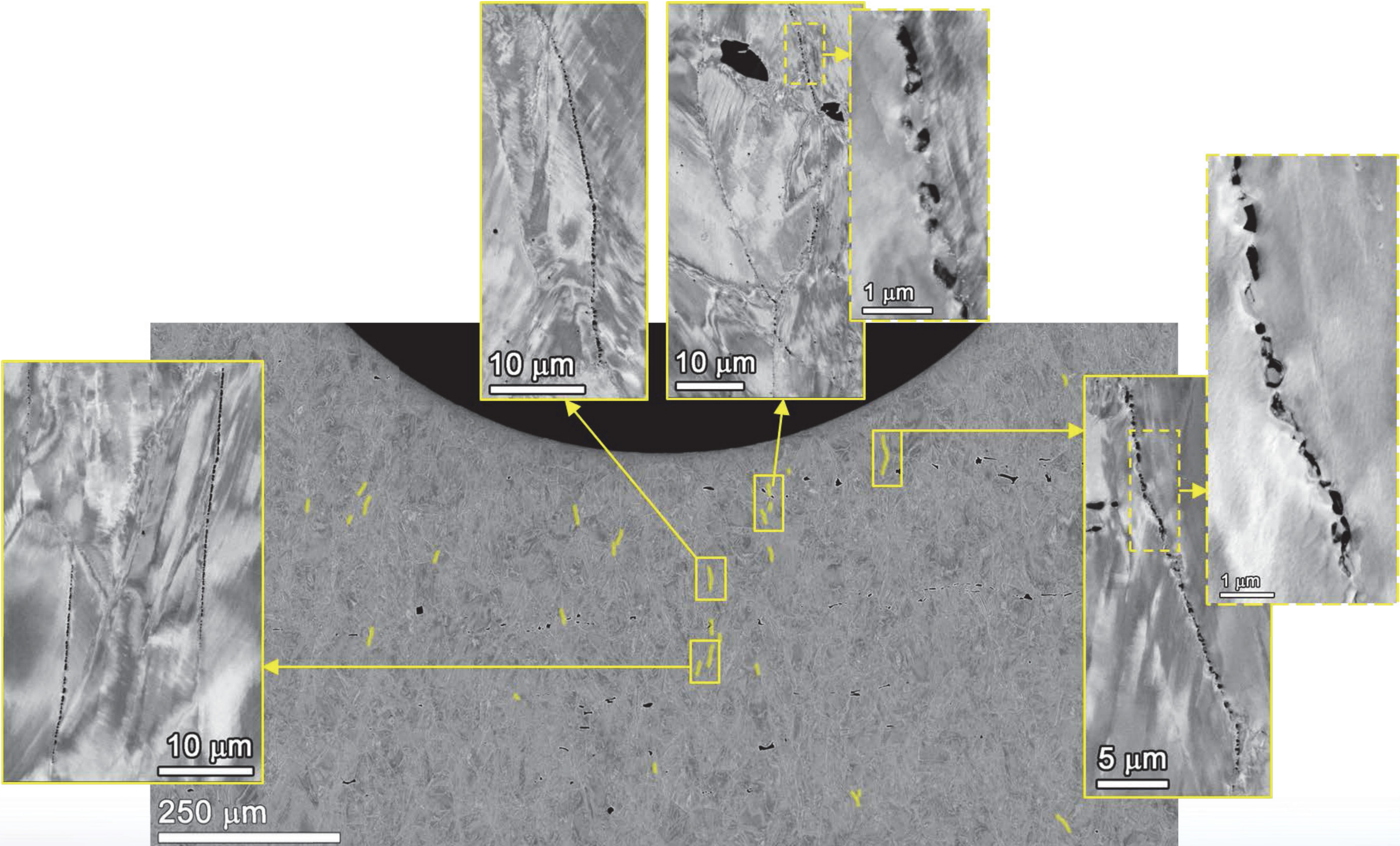
C703 31%CF Valinox CRDM

Blunt Notch – Typical Crack Morphology on Cross-Section (C707, 21%CF Valinox CRDM)



- Small, shallow IG cracks identified to depths <~3 µm.
- Creep cavities were observed ahead of some crack/oxidation tips.
- Creep cavities were also found close to the notch surface along some intact grain boundaries.

Distribution of Creep Cavities in Blunt Notch C704 31%CF Sumitomo CRDM



Similar distribution of grain boundary creep cavities in the 31%CF Sumitomo CRDM as for the Valinox CRDM.

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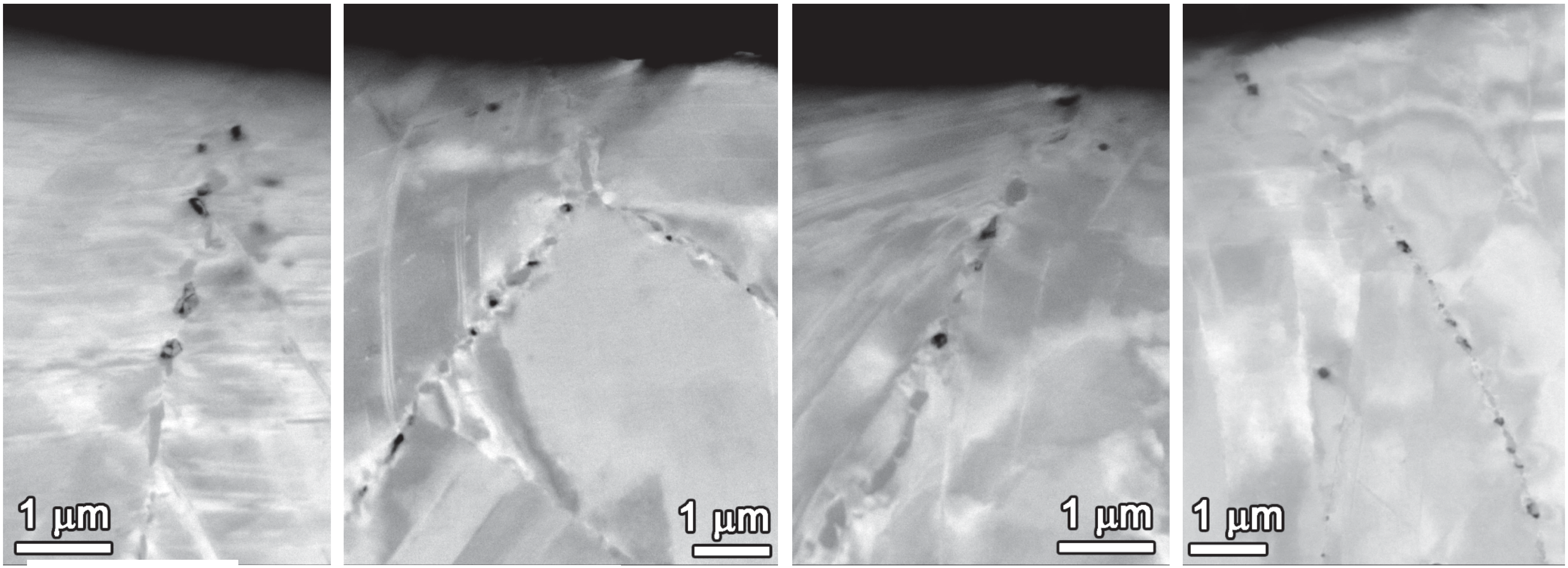
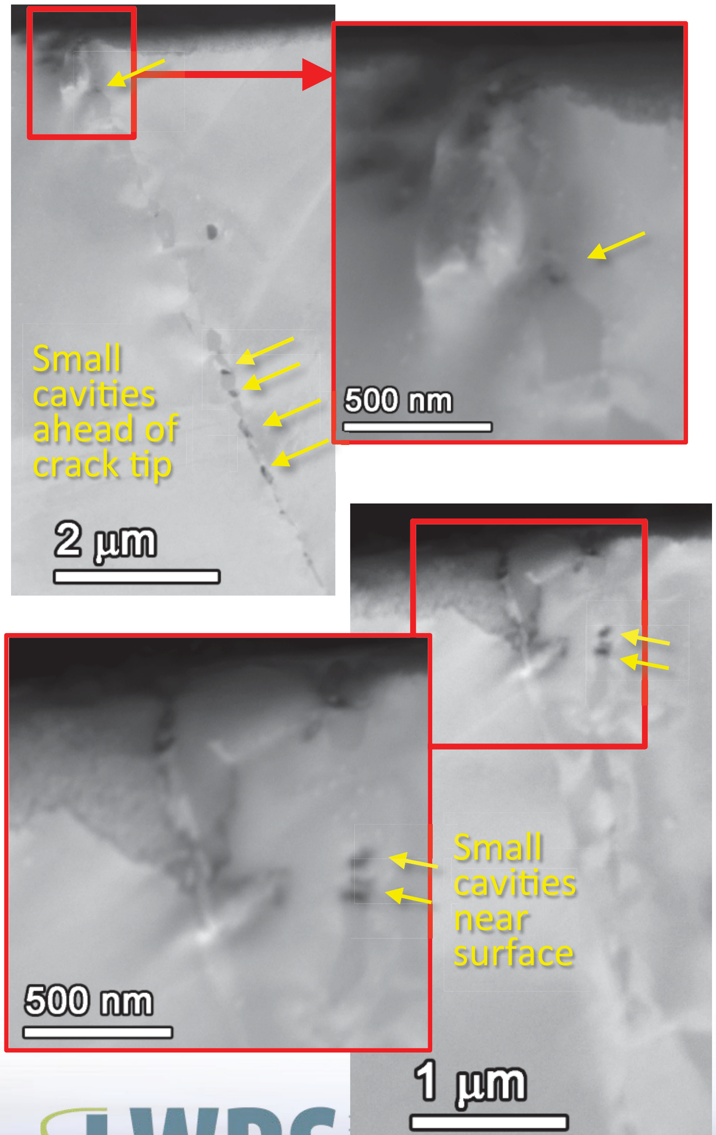
Blunt Notch C704, 31%CF Sumitomo CRDM – Near-Surface Grain Boundary Microstructures

- Creep cavities were very found close to the notch surface along a number of grain boundaries without evidence of IG crack nucleation.

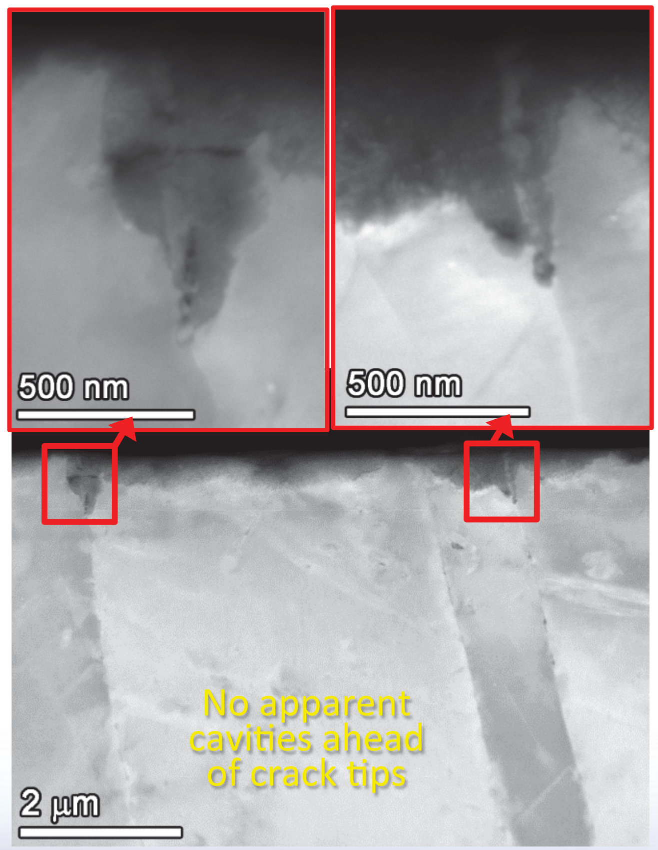


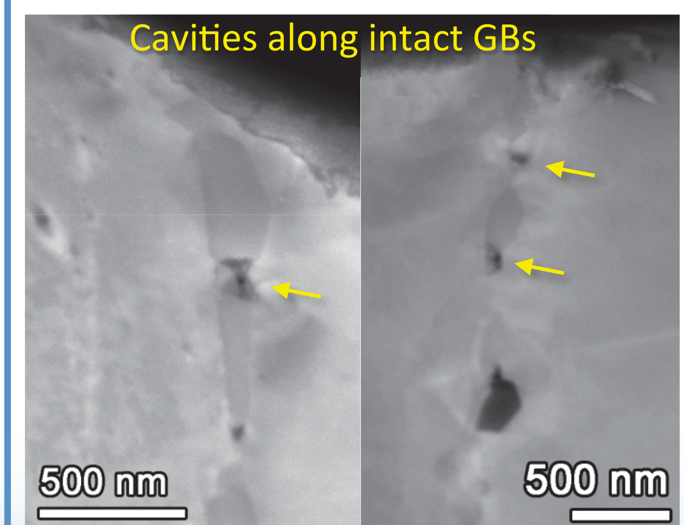
Blunt Notch – Typical Crack Morphology on Cross-Section (C708, 21%CF Sumitomo CRDM)

Short cracks: Depth < 1 μm



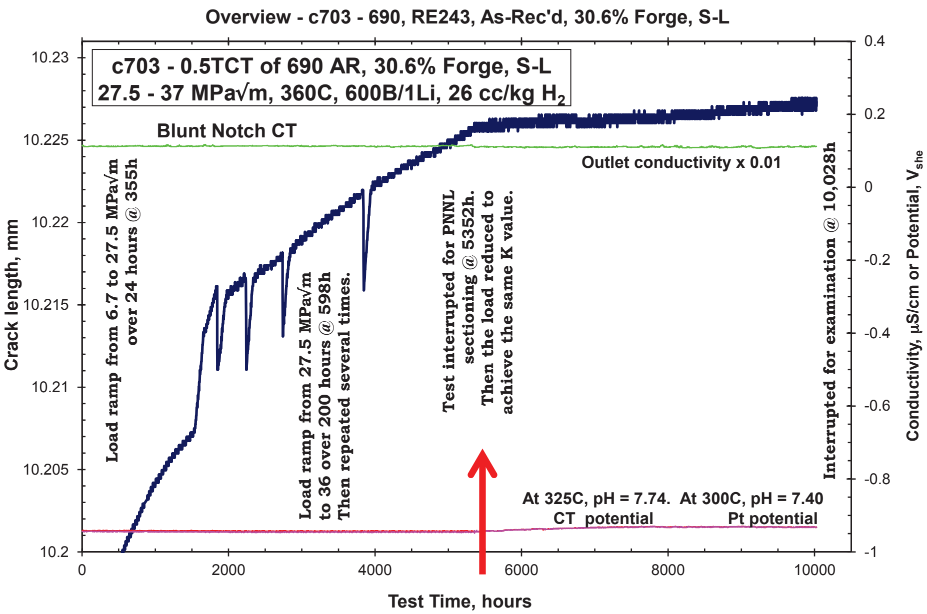
- Low density of very shallow IG cracks.
- Creep cavities were observed ahead of some crack tips and were also found close to the notch surface along some intact grain boundaries.
- Creep cavities are smaller and more discrete compared to those in C703 & C704.

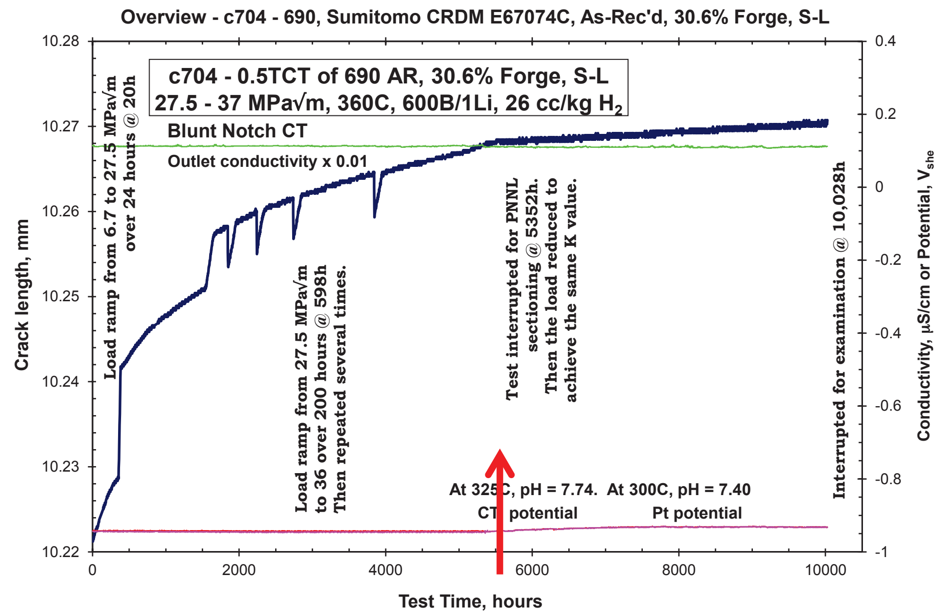


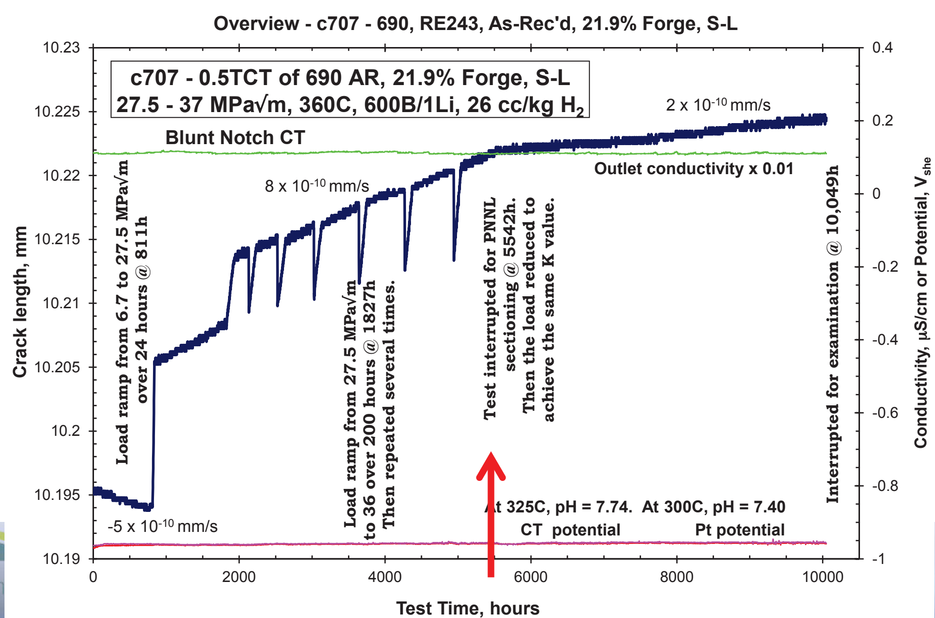


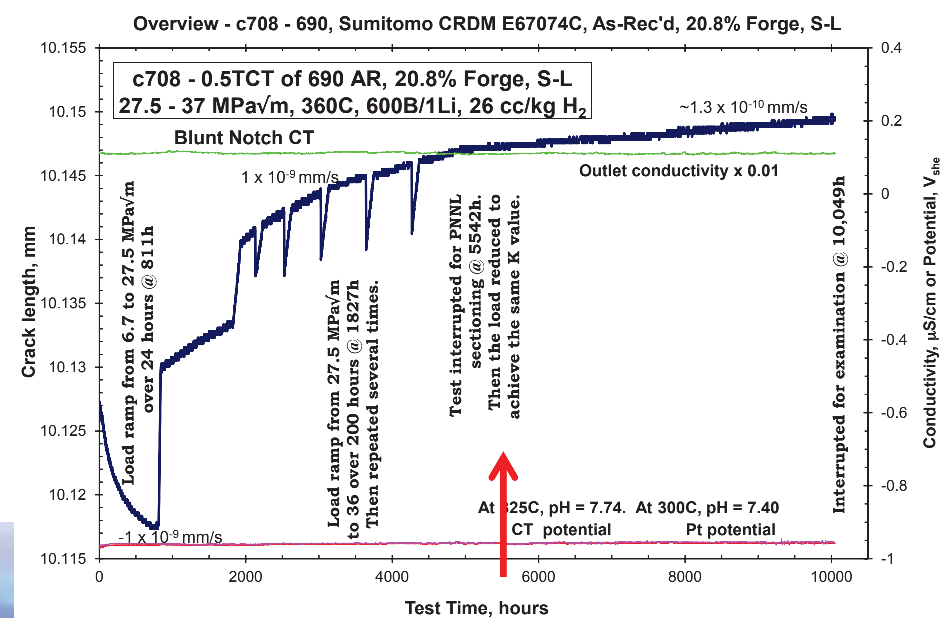
Phase 2 Blunt Notch SCC Initiation Tests

- 0 - 5,325 h: 4-6 loading ramps at K = 27.5 to 36 MPa√m + ~500 h hold
- 5,325 - 10,000 h: Constant load at K = 37 MPa√m









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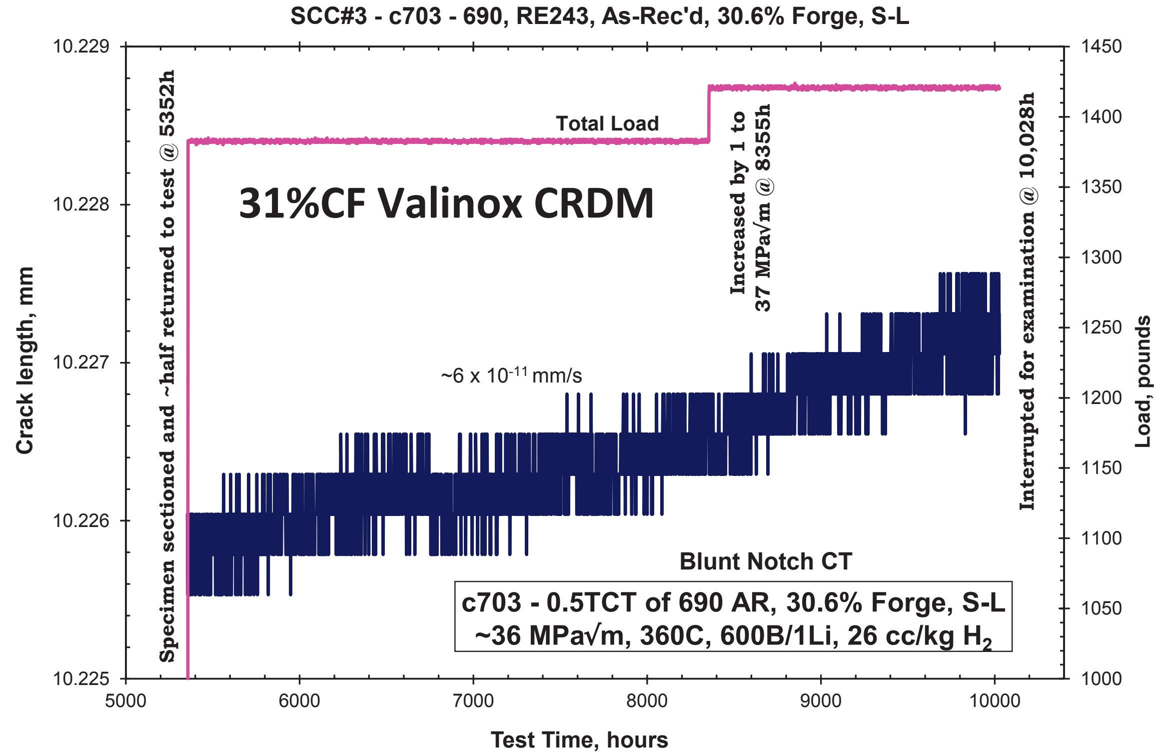
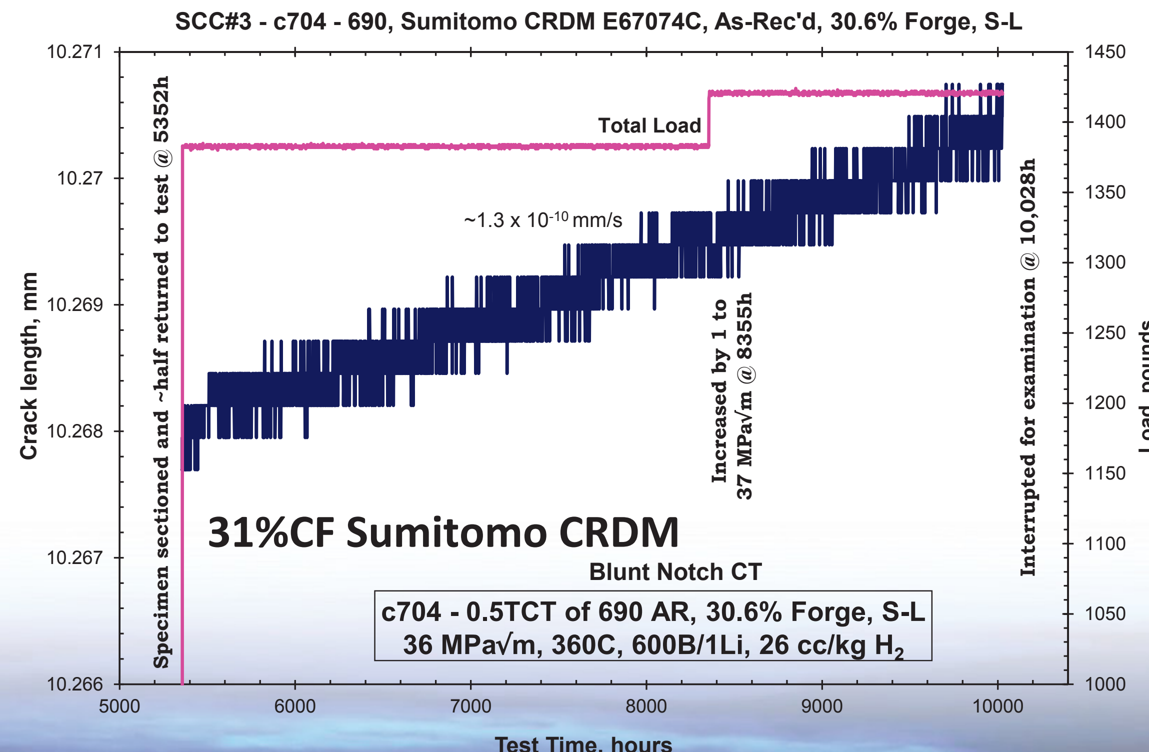
Continued Phase 2 Blunt Notch SCC Initiation Tests at GEG

- 0 - 5,325h: 4-6 loading ramps at K = 27.5 to 36 MPa√m + ~500h hold
- 5,325 - 10,000h: Constant load at K = 36-37 MPa√m
- Extremely low DCPD evolution at constant load
- No indication of crack nucleation or growth of existing small cracks.





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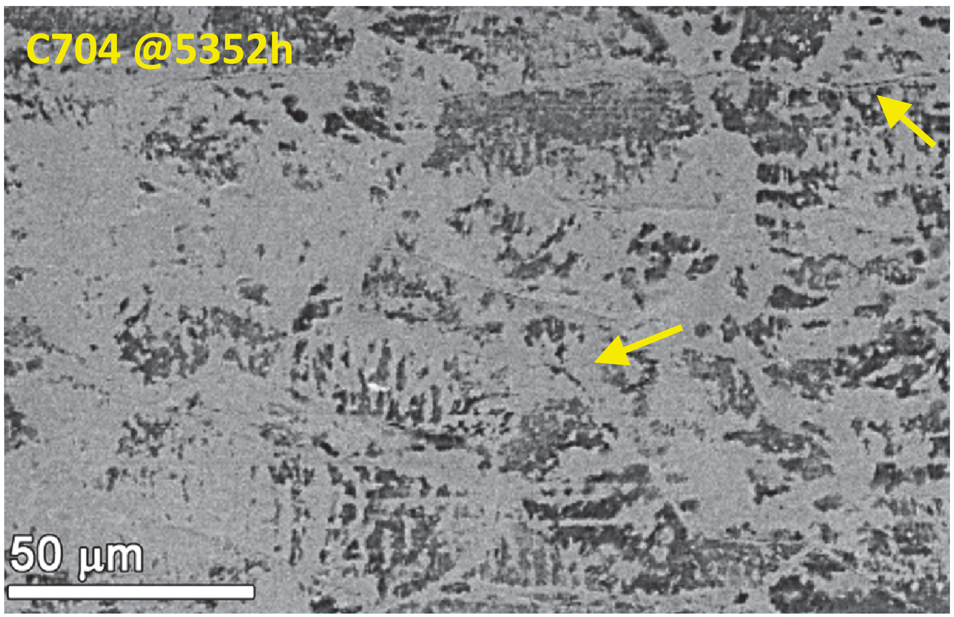
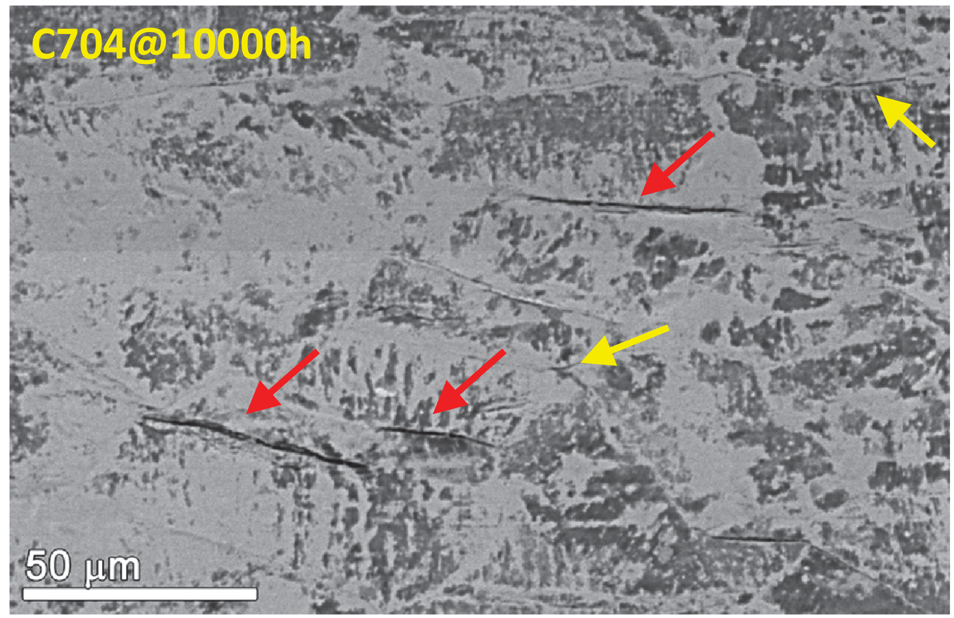
SEM Surface Examinations Underway on Blunt Notch Specimens after 10,000 h Exposure

- SEM mapping is being performed for direct comparisons of current surface microstructures to those obtained after 5352 h.
- Preliminary results do not reveal any significant change in the preexisting IG cracks, nor definite formation of new cracks, for the Valinox specimens.
- However, the 31%CF Sumitomo CRDM specimen C704 appears to show several small IG cracks have developed under constant load as illustrated below. May result from growth and coalescence of very small cracks.





Summary of Alloy 690 Blunt Notch Test Results

- IG crack initiation observed on highly cold-worked alloy 690 CRDM materials when minor plastic strain is produced during cyclic loading, much greater cracking for 31%CF versus 21%CF materials.
- Grain boundary cavity formation appears to be directly involved in the nucleation of small IG “precursor” cracks at the blunt notch surfaces.
- Dynamic straining during cyclic loading appears to responsible for grain boundary cavitation and cracking.
- It remains unclear whether this precursor IG damage will occur under constant load conditions, preliminary exams suggest evolution of existing IG surface cracks may occur.

Outline

- Blunt-Notch SCC Initiation Tests
  - Phase 1: 31%CF Alloy 690 CRDM Heats
  - Phase 2: 31%CF and 21%CF Alloy 690 CRDM Heats
  - Characterization of IG Crack Nucleation & Growth
- Tensile Constant Load SCC Initiation Tests
  - Long-Term Exposures of 12%CF, 20%CF and 30%CF Alloy 690 CRDM and Plate Heats
  - Surface and Near Surface Characterization of IG Damage Evolution after Test Time of ~1 year

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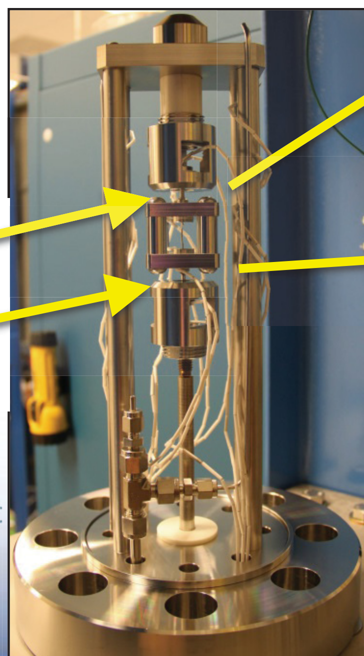
Stress Corrosion Crack Initiation Testing in LWR Environments

State-of-the-art SCC initiation test systems assembled with in-situ DCPD crack detection at micrometer resolution.

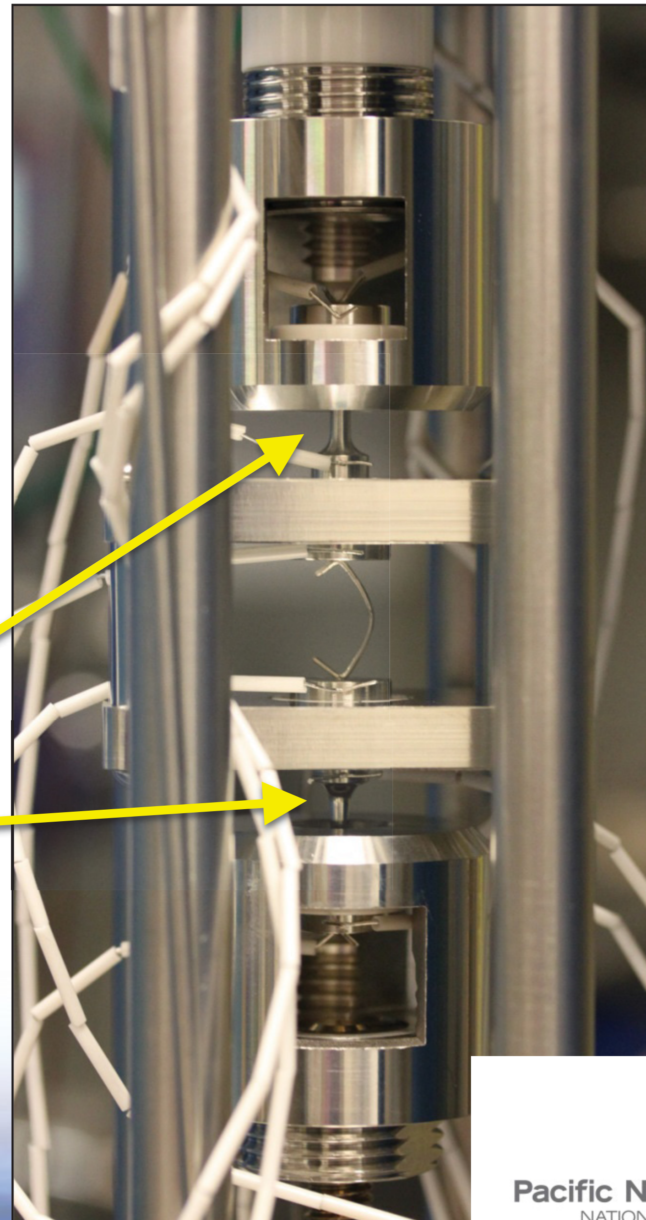
Two high-temperature, high-pressure autoclave systems with 6 fully instrumented specimens and one system for 36 specimens with up to 20 instrumented.

1.2" Tall SCC Initiation Specimen

2-6 Specimen SCC Initiation System



2-6 Specimen SCC Initiation System

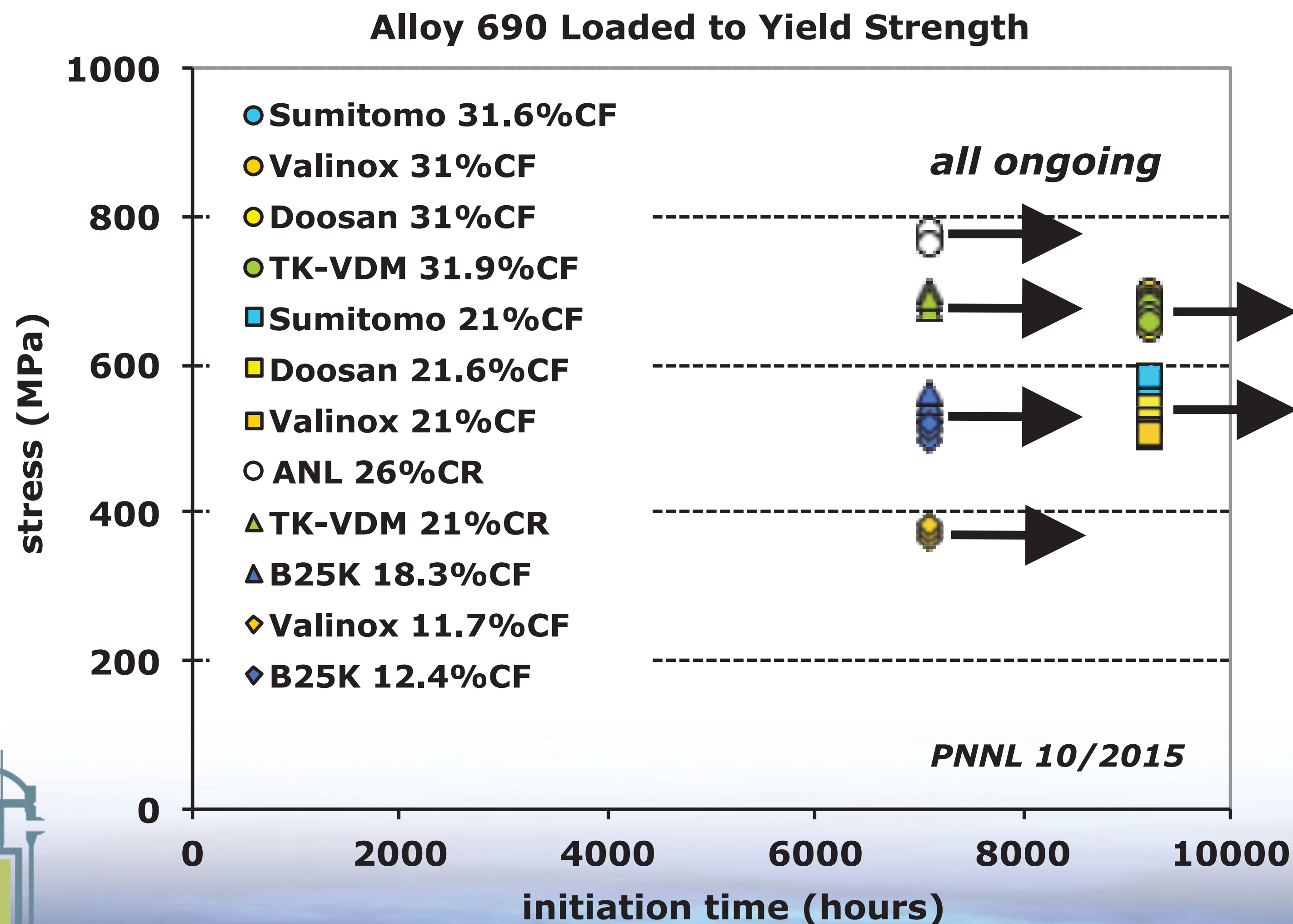


36 Specimen SCC Initiation System

Pacific Northwest
NATIONAL LABORATORY

Status of 36 Specimen SCC Initiation Test on Cold Worked Alloy 690

Constant load tests have reached 0.8-1.1 year exposure for ~12%, ~20% and ~30%CF alloy 690 CRDM and plate materials in 360C simulated PWR primary water. No evidence for crack initiation from DCPD, test stopped in September and surface exams are underway on selected specimens.



Constant Tensile Load SCC Initiation Tests

Constant Load Alloy 690 SCC Initiation Tests in LWRS System 1: First Set of 21 Specimens after ~5750, 7050, 9220 hour Exposure Time

Specimen	Material	Heat Number	Material Condition	Surface Condition	Appl. Stress, MPa	DCPD Strain Rate, %ε/h
IN024 (CL)	**Sumitomo CRDM**	**EC7074C**	**TT + 21%CF**	**1 µm Polish**	**575**	**$1.1x10^{-4}$; $1.4x10^{-4}$; $7x10^{-5}$**
IN025 (CL)	Sumitomo CRDM	EC7074C	TT + 21%CF	Ground: C Finish	575	
IN026 (CL)	Sumitomo CRDM	EC7074C	TT + 21%CF	Ground: C Finish	575	
IN027 (CL)	**Valinox CRDM**	**RE243**	**TT + 21%CF**	**1 µm Polish**	**510**	**$1.0x10^{-4}$; $8x10^{-5}$; $4x10^{-5}$**
IN028 (CL)	Valinox CRDM	RE243	TT + 21%CF	Ground: C Finish	510	
IN029 (CL)	Valinox CRDM	RE243	TT + 21%CF	Ground: C Finish	510	
IN030 (CL)	**Doosan CRDM**	**133454**	**TT + 21.6%CF**	**1 µm Polish**	**540**	**$1.1x10^{-4}$; $6x10^{-5}$; $4x10^{-5}$**
IN031 (CL)	Doosan CRDM	133454	TT + 21.6%CF	Ground: C Finish	540	
IN032 (CL)	Doosan CRDM	133454	TT + 21.6%CF	Ground: C Finish	540	
IN033 (CL)	**Sumitomo CRDM**	**EC7074C**	**TT + 31%CF**	**1 µm Polish**	**690**	**$1.6x10^{-4}$; $1.2x10^{-4}$; $1.2x10^{-4}$**
IN034 (CL)	Sumitomo CRDM	EC7074C	TT + 31%CF	Ground: C Finish	690	
IN035 (CL)	**Sumitomo CRDM**	**EC7074C**	**TT + 31%CF**	**Ground: C Finish**	**690**	
IN036 (CL)	**Valinox CRDM**	**RE243**	**TT + 31%CF**	**1 µm Polish**	**700**	**$1.8x10^{-4}$; $2.8x10^{-4}$; $9x10^{-5}$**
IN037 (CL)	Valinox CRDM	RE243	TT + 31%CF	Ground: C Finish	700	
IN038 (CL)	Valinox CRDM	RE243	TT + 31%CF	Ground: C Finish	700	
IN039 (CL)	**Doosan CRDM**	**133454**	**TT + 31%CF**	**1 µm Polish**	**665**	**- - - ; $1.6x10^{-4}$; ~0**
IN040 (CL)	Doosan CRDM	133454	TT + 31%CF	Ground: C Finish	665	
IN041 (CL)	Doosan CRDM	133454	TT + 31%CF	Ground: C Finish	665	
IN042 (CL)	**TK-VDM Plate**	**114092**	**TT + 31.9%CF**	**1 µm Polish**	**680**	**$1.8x10^{-4}$; $1.7x10^{-4}$; $1.2x10^{-4}$**
IN043 (CL)	TK-VDM Plate	114092	TT + 31.9%CF	Ground: C Finish	680	
IN044 (CL)	TK-VDM Plate	114092	TT + 31.9%CF	Ground: C Finish	680	

Servo displacement of ~0.005 µm/h from 7000-9220 hours

LWRS
Light Water Reactor Sustainability

Highlighted specimens: ongoing surface exams by SEM and FIB.

Constant Tensile Load SCC Initiation Tests

Constant Load Alloy 690 SCC Initiation Tests in LWRS System 1: Second Set of 15 Specimens after ~3610, 4910 and 7100 hour Exposure Time

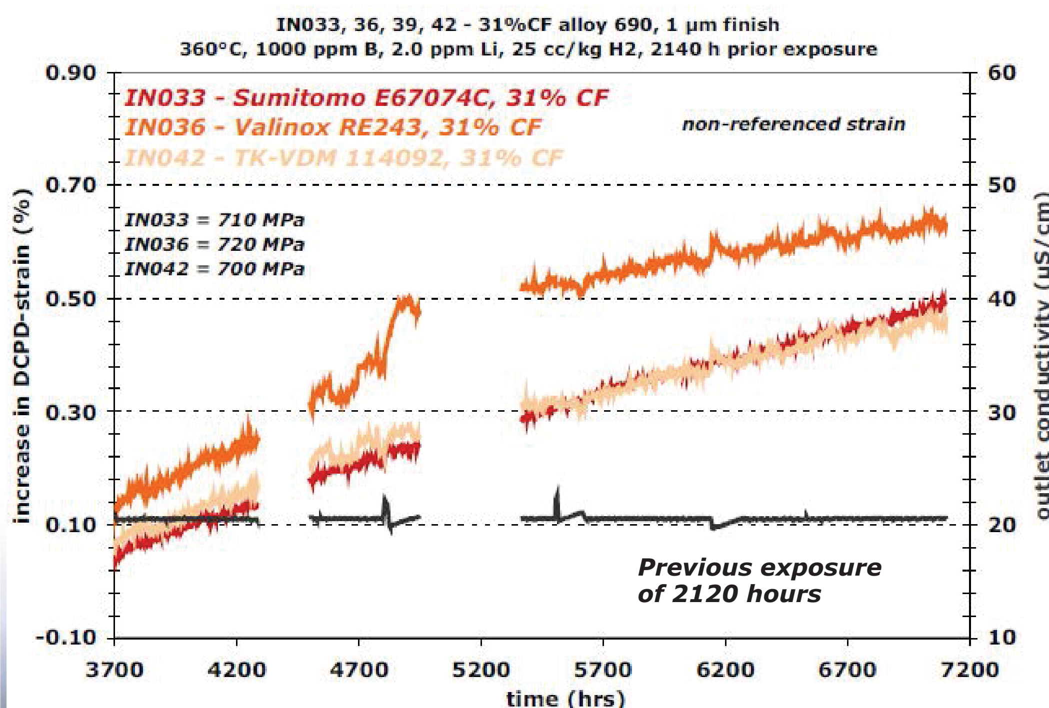
Specimen	Material	Heat Number	Material Condition	Surface Condition	Applied Stress, MPa	Non-Ref DCPD, %ε/h
IN053 (CL)	**ANL Flat Bar**	**NX3297HK12**	**MA + 26%CR**	**1 μm Polish**	**775**	**$1.6x10^{-4}$; $1x10^{-4}$; $9x10^{-5}$**
IN054 (CL)	ANL Flat Bar	NX3297HK12	MA + 26%CR	1 μm Polish	775	
IN055 (CL)	ANL Flat Bar	NX3297HK12	MA + 26%CR	Ground: C Finish	775	
IN056(CL)	**GE B25K Bar**	**B25K**	**MA + 18.3%CF**	**1 μm Polish**	**550**	**$1.6x10^{-4}$; $1x10^{-4}$; $8x10^{-5}$**
IN057(CL)	GE B25K Bar	B25K	MA + 18.3%CF	1 μm Polish	550	
IN058 (CL)	GE B25K Bar	B25K	MA + 18.3%CF	Ground: C Finish	550	
IN059 (CL)	**TK-VDM Plate**	**114092**	**TT + 21%CR**	**1 μm Polish**	**690**	**$1.6x10^{-4}$; $1x10^{-4}$; $9x10^{-5}$**
IN060 (CL)	TK-VDM Plate	114092	TT + 21%CR	1 μm Polish	690	
IN061 (CL)	TK-VDM Plate	114092	TT + 21%CR	Ground: C Finish	690	
IN062 (CL)	**GE B25K Bar**	**B25K**	**MA + 12.4%CF**	**1 μm Polish**	**510**	**$1.3x10^{-4}$; $8x10^{-5}$; $7x10^{-5}$**
IN063 (CL)	GE B25K Bar	B25K	MA + 12.4%CF	1 μm Polish	510	
IN064 (CL)	GE B25K Bar	B25K	MA + 12.4%CF	Ground: C Finish	510	
IN065 (CL)	**Valinox CRDM**	**RE243**	**TT + 11.7%CF**	**1 μm Polish**	**365**	**$1.0x10^{-4}$; $7x10^{-5}$; $5x10^{-5}$**
IN066 (CL)	Valinox CRDM	RE243	TT + 11.7%CF	1 μm Polish	365	
IN067 (CL)	Valinox CRDM	RE243	TT + 11.7%CF	Ground: C Finish	365	

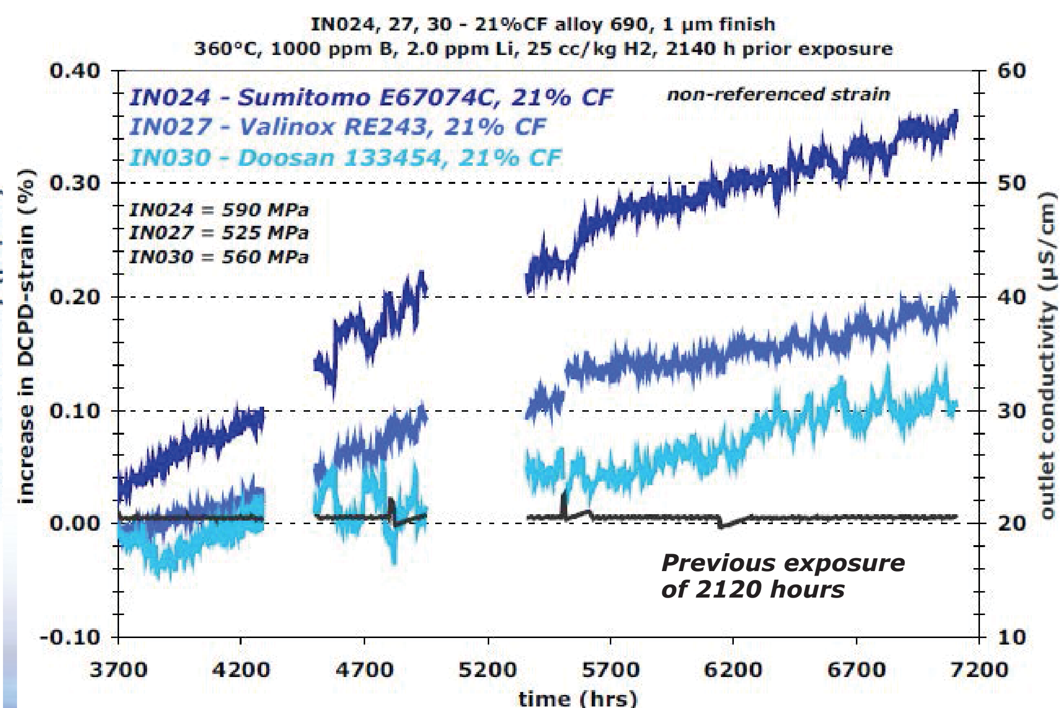
Servo displacement of ~0.005 μm/h from 5000-7100 hours

Highlighted specimens: ongoing surface exams by SEM and FIB.

Constant Tensile Load SCC Initiation Tests

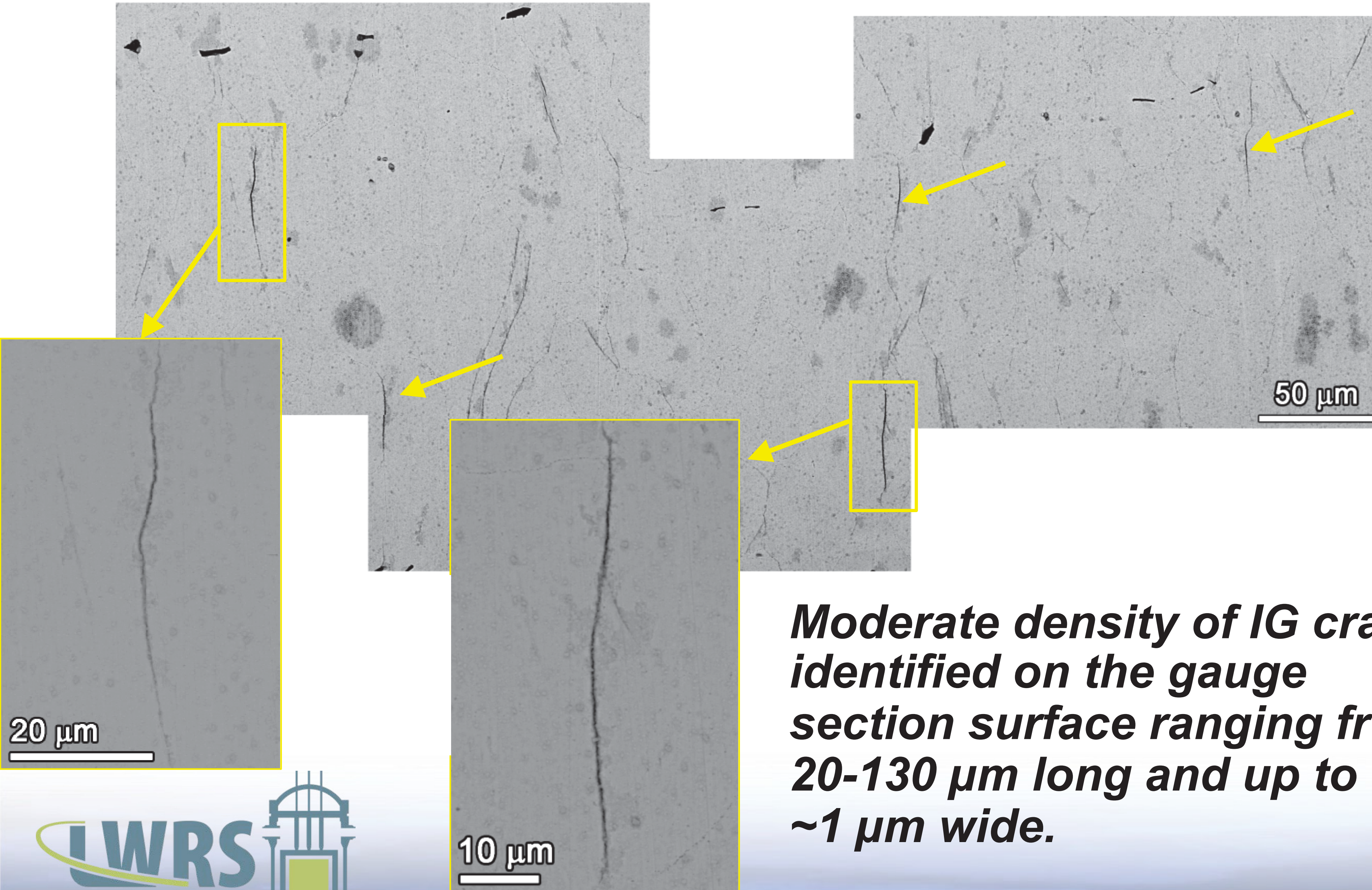
Spec	Material & Condition	Surface	σ (MPa)	SEM Observations
IN033	Sumitomo CRDM, TT+31%CF	1 µm	690	Many small IG cracks
IN035	Sumitomo CRDM, TT+31%CF	Ground	690	Few small cracks
IN024	Sumitomo CRDM, TT+21%CF	1 µm	575	Isolated small IG cracks
IN036	Valinox CRDM, TT+31%CF	1 µm	700	Many small IG cracks
IN027	Valinox CRDM, TT+21%CF	1 µm	510	No cracks
IN065	Valinox CRDM, TT+12%CF	1 µm	365	No cracks





SEM Examinations of Surface Damage

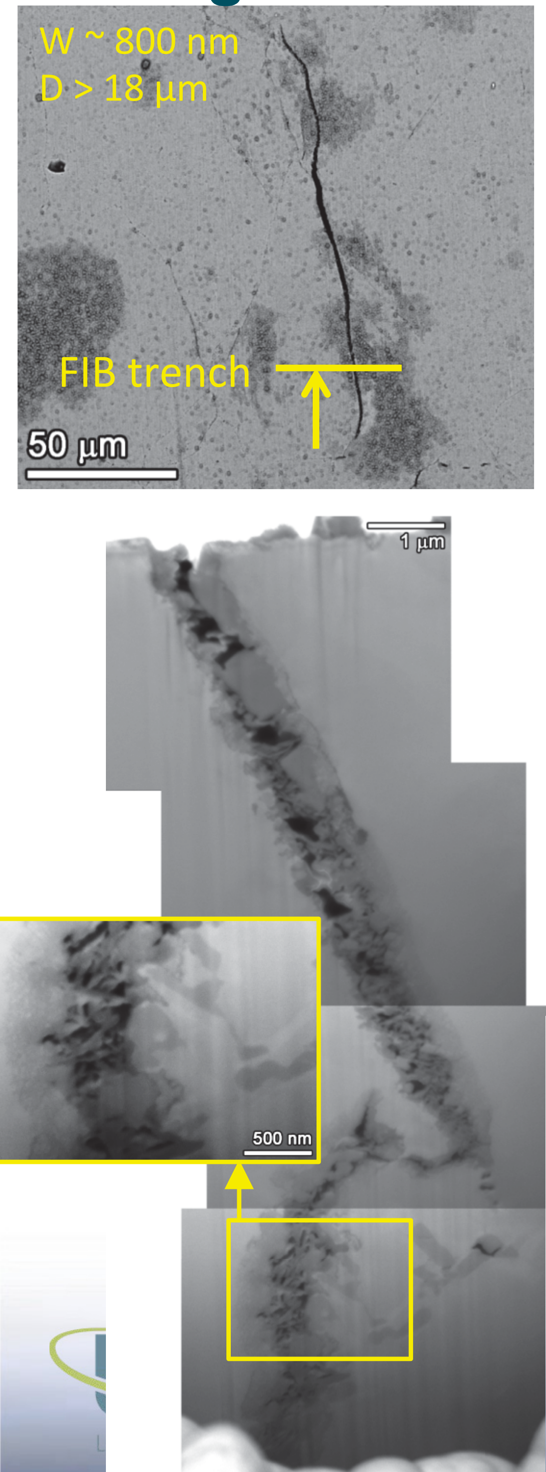
IN033 31% CF Sumitomo CRDM



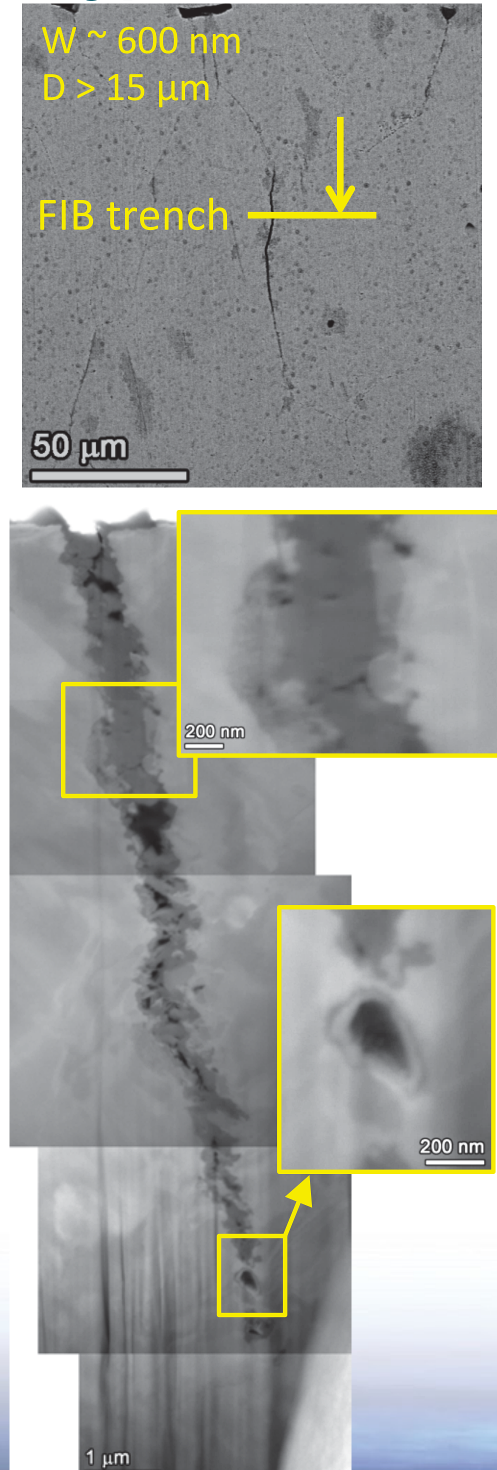
Moderate density of IG cracks identified on the gauge section surface ranging from 20-130 μm long and up to ~1 μm wide.

Light Water Reactor Sustainability

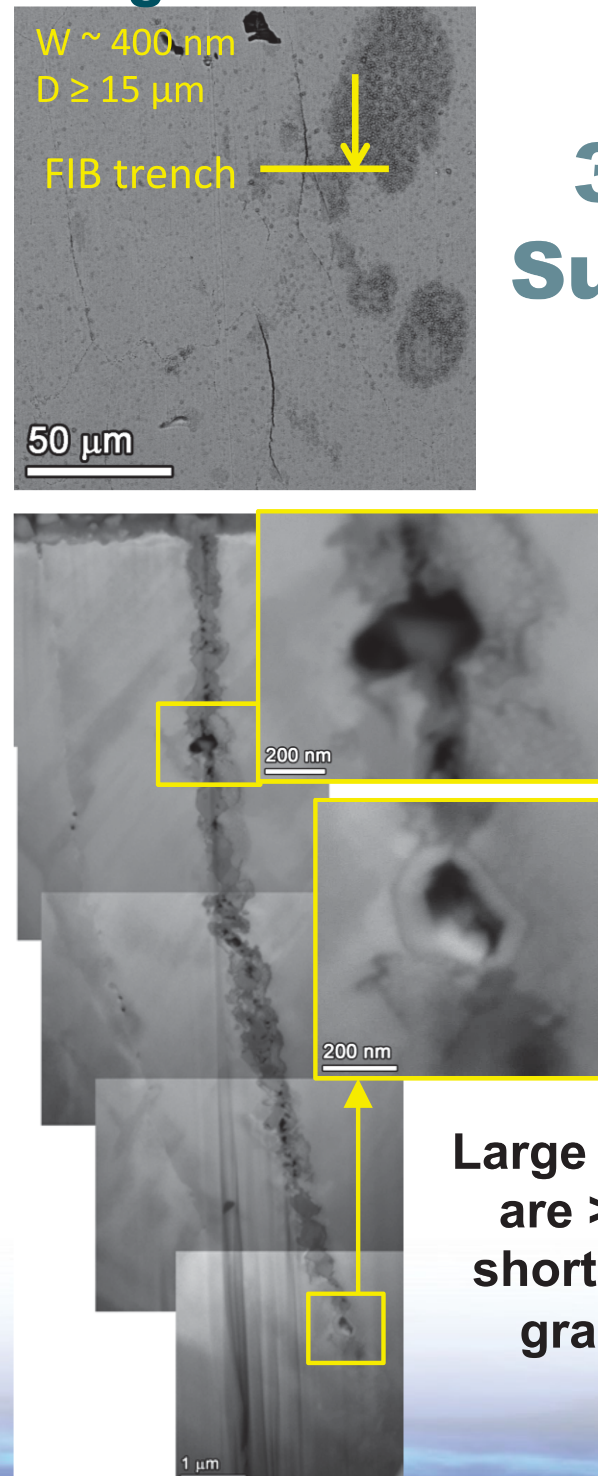
Large crack



Typical crack



Tight crack

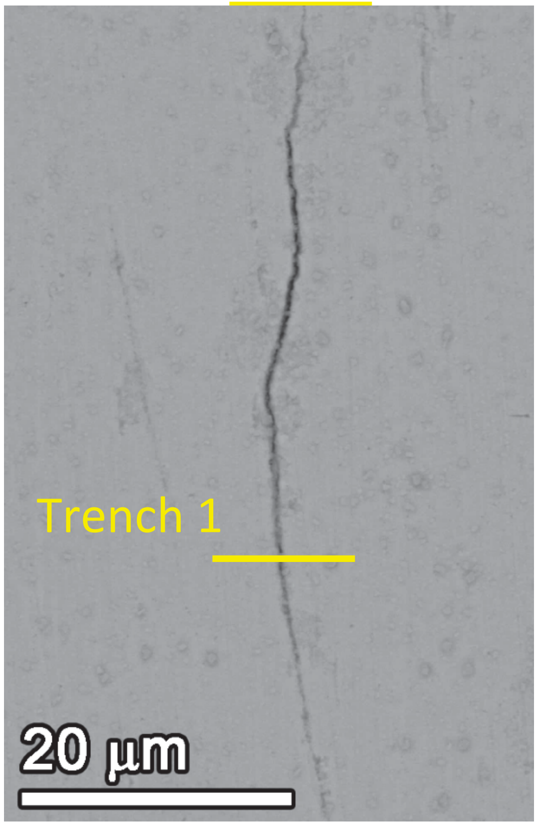


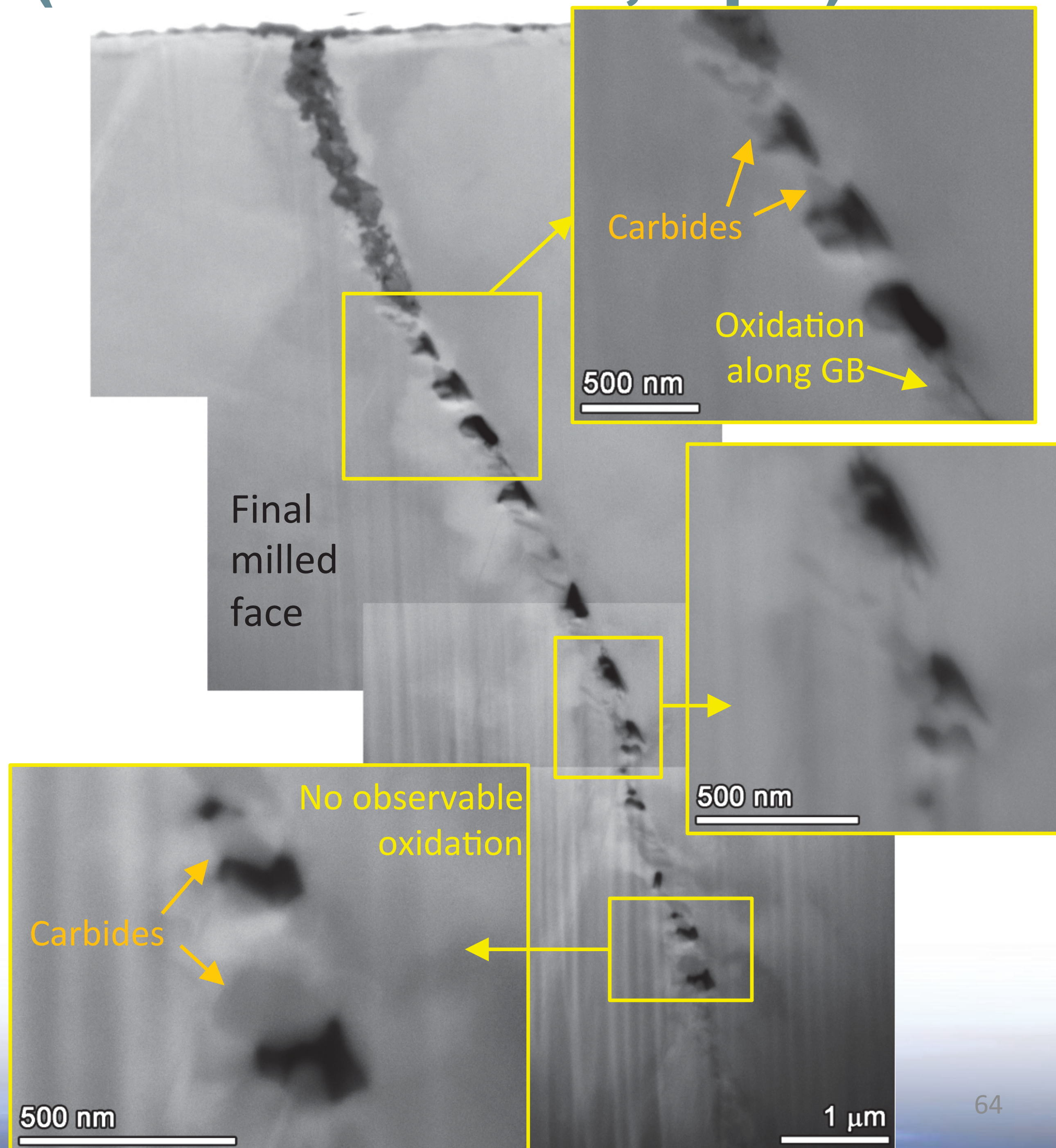
IN033
31%CF
Sumitomo
CRDM

FIB trenches, SEM of cracks in cross-section

Large surface cracks are >15 µm deep, short cracks end in grain boundary cavities.

IG Crack and Cavity Morphology in IN033 (Sumitomo 31%CF, 1 μm)





- IG oxide filled crack reaching >15 μm deep.
- Grain boundary cavities found ahead of the crack/oxidation tip.

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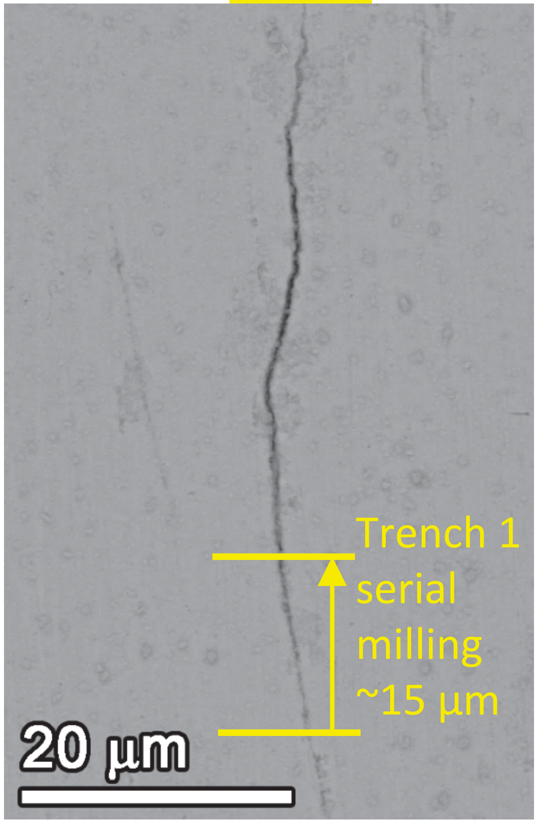
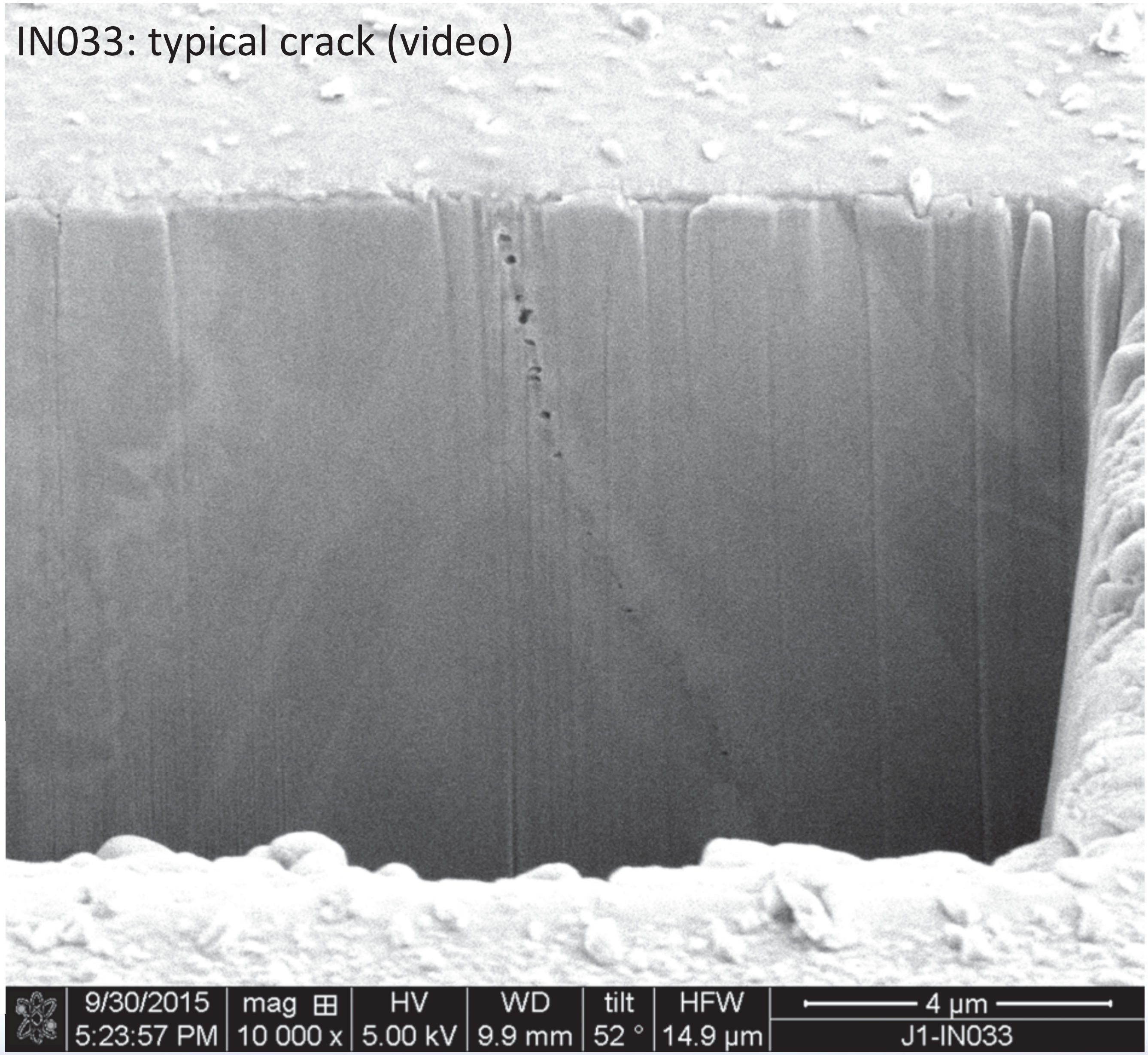
IN033 (Sumitomo 31%CF, 1 μm) FIB Serial Milling: Grain Boundary Cavities and IG Crack





- IG oxide filled crack reaching >15 μm deep.
- Grain boundary cavities found ahead of the crack/oxidation tip.

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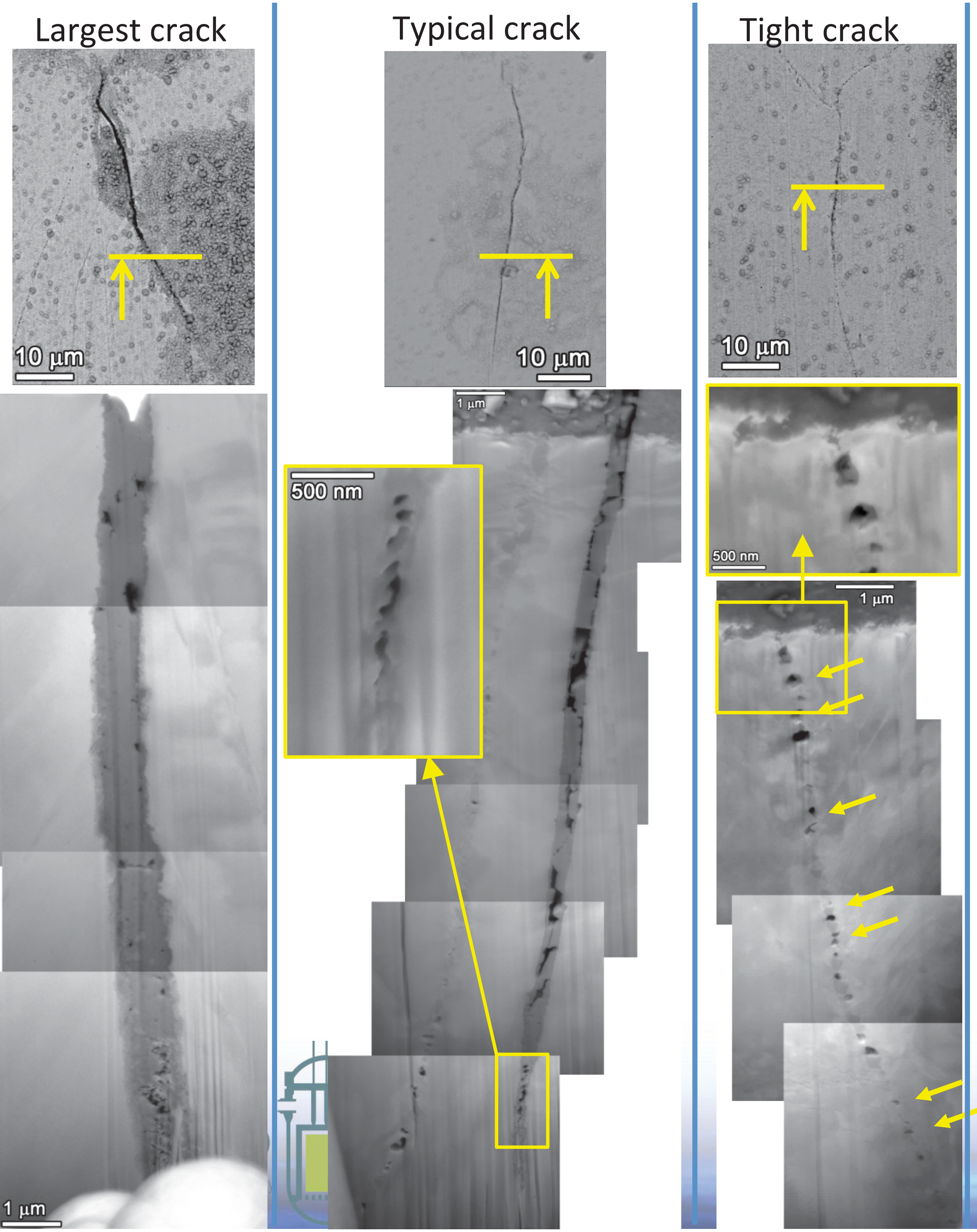
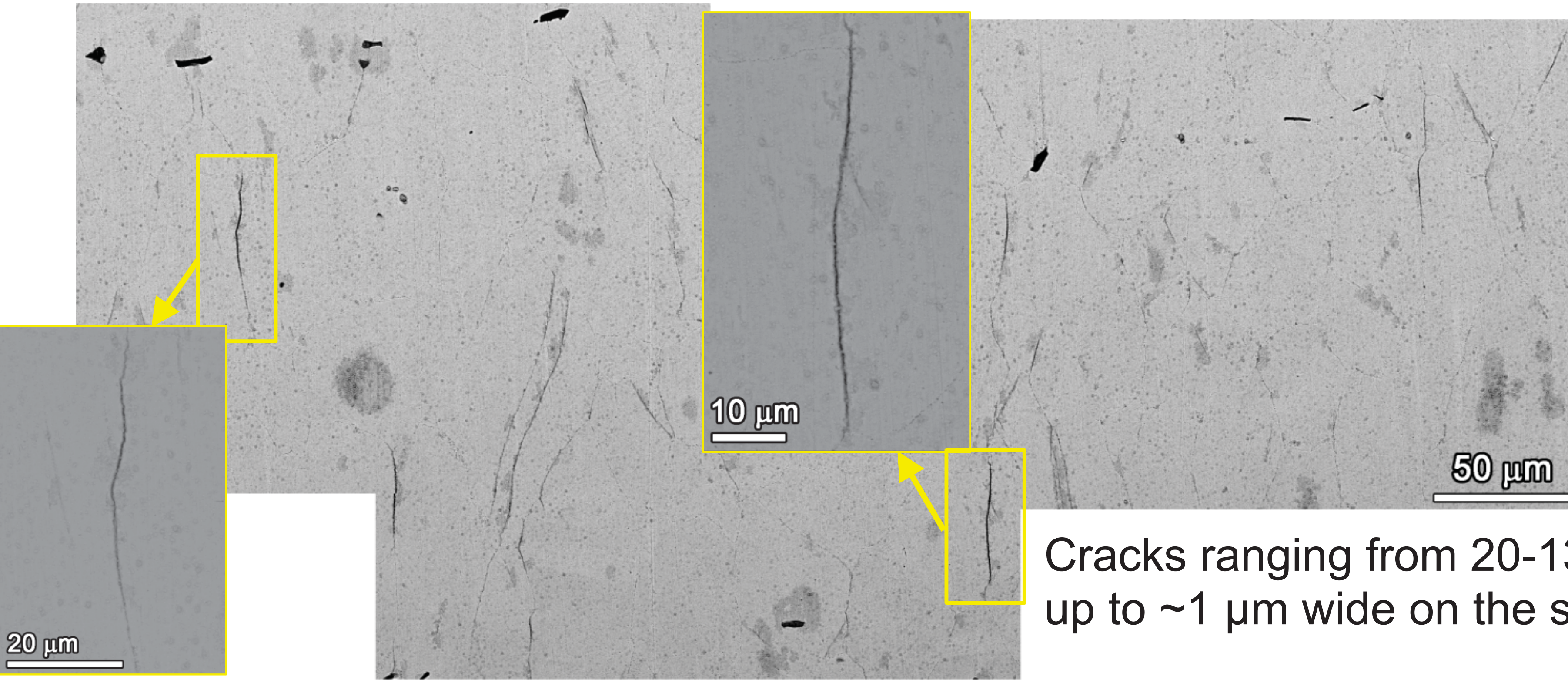


IN036 31%CF Valinox CRDM

FIB + SEM Examples of IG Cracks

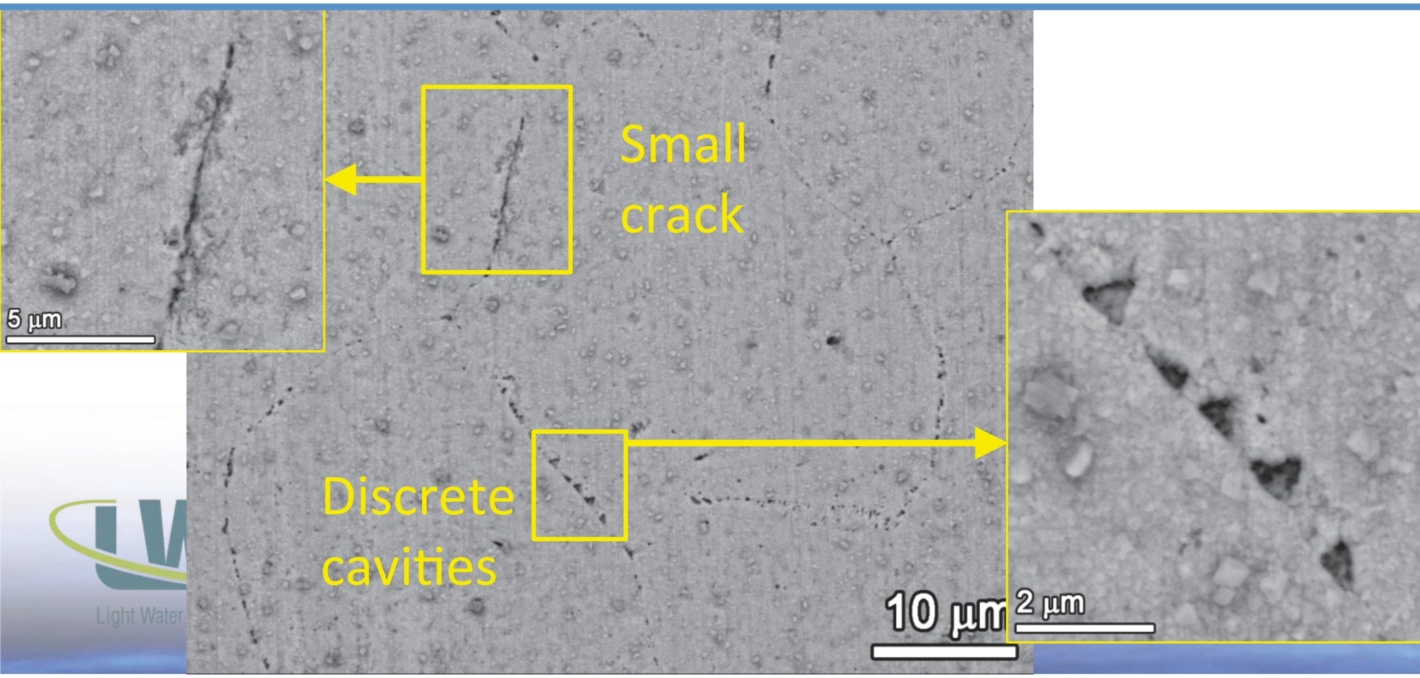
- Larger surface cracks much greater than 15 μm in depth.
- Grain boundary cavities typically found leading shallow cracks.
- Grain boundary cavity formation appears to be a precursor to IG crack nucleation and growth of short cracks.

Surface Damage Morphology – Sumitomo CRDM



31% CF (IN033)

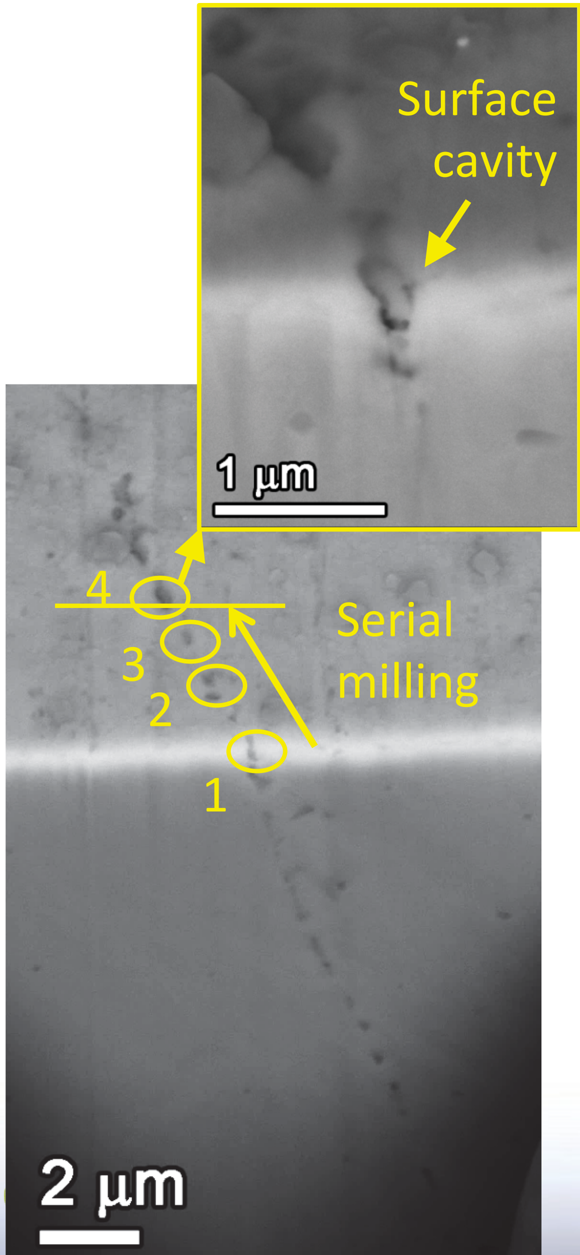
Cracks ranging from 20-130 µm long, up to ~1 µm wide on the surface

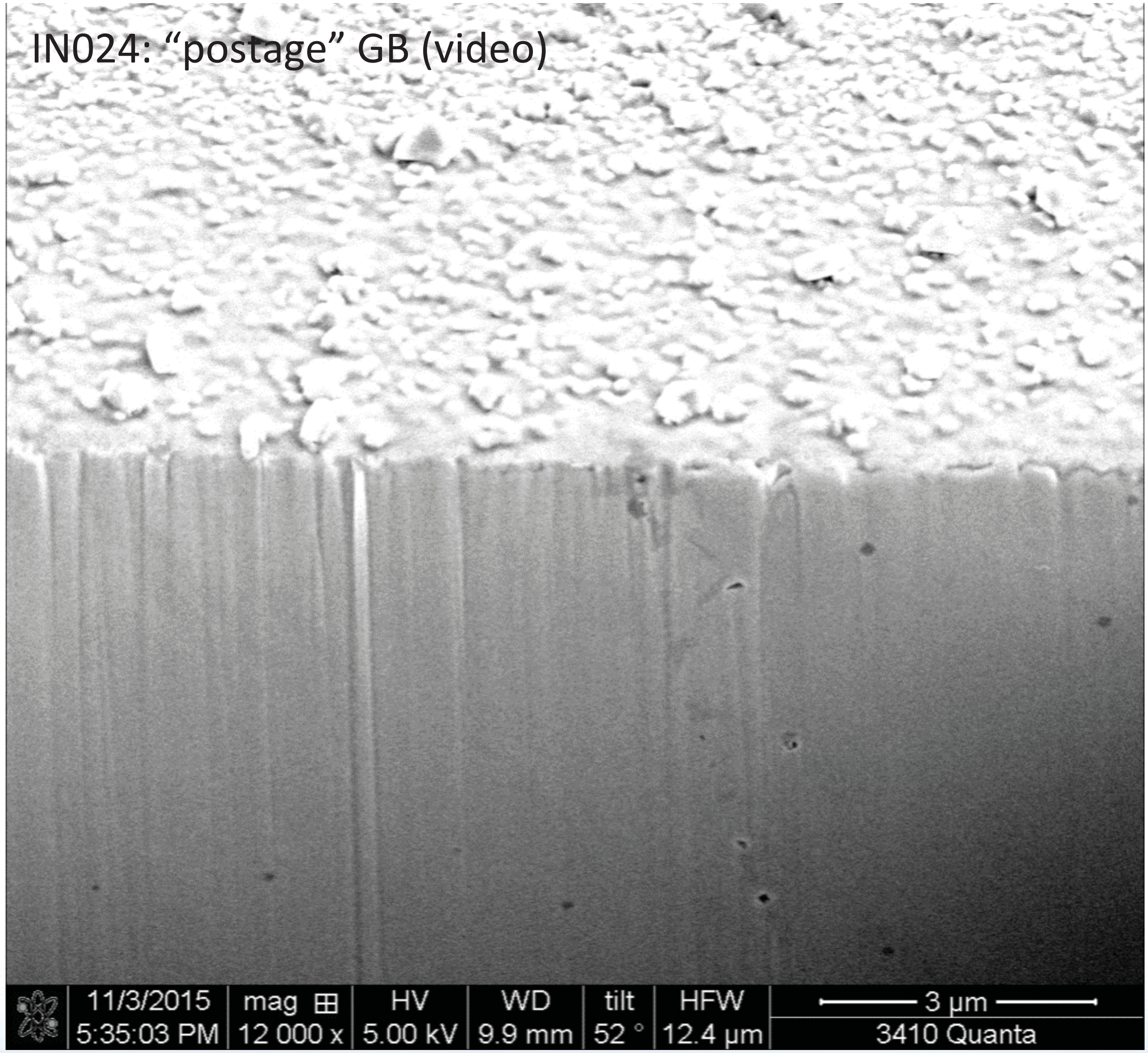


21% CF (IN024)

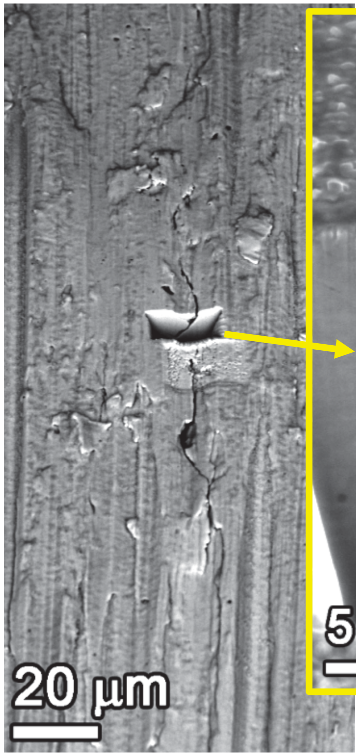
Only a few short, shallow cracks observed and “postage” damage (discrete cavities) along grain boundaries

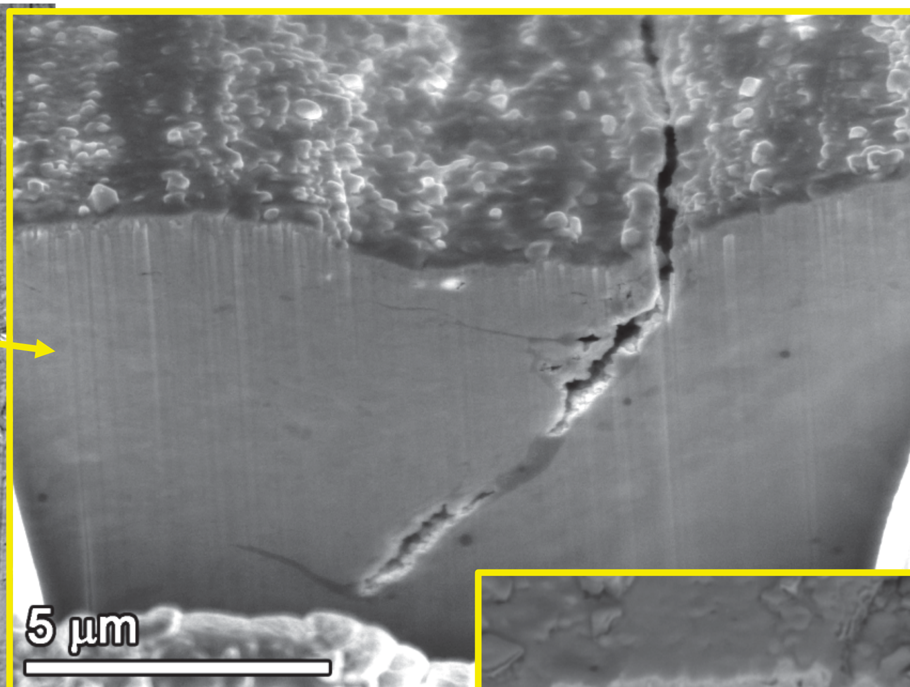
IN024 (21%CF Sumitomo CRDM, 1 µm) FIB Serial Milling: Surface Cavity Distribution



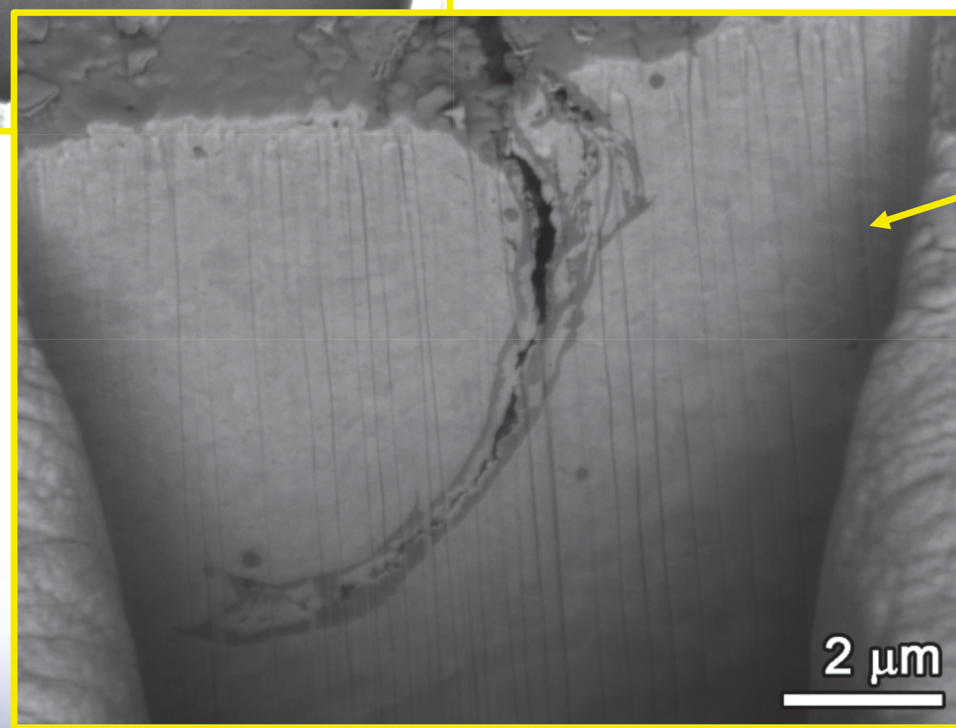


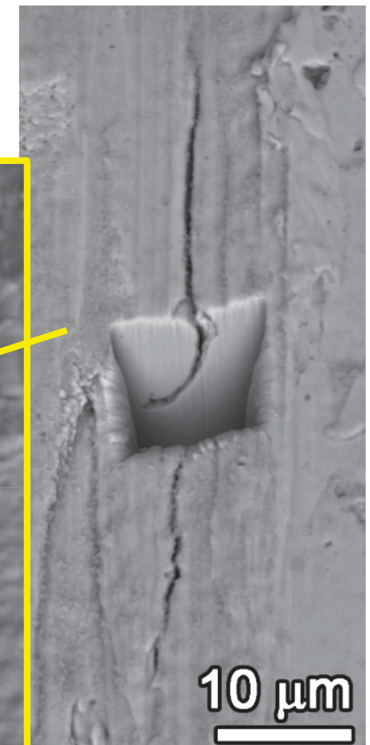
IN035 (Sumitomo 31%CF, "C" finish) FIB+SEM: Near Surface Crack Morphology





- Shallow cracks are confined to the heavily deformed, nanocrystalline layer (<10 µm deep) due to grinding.
- No evidence of cavity formation in the surface damage layer.

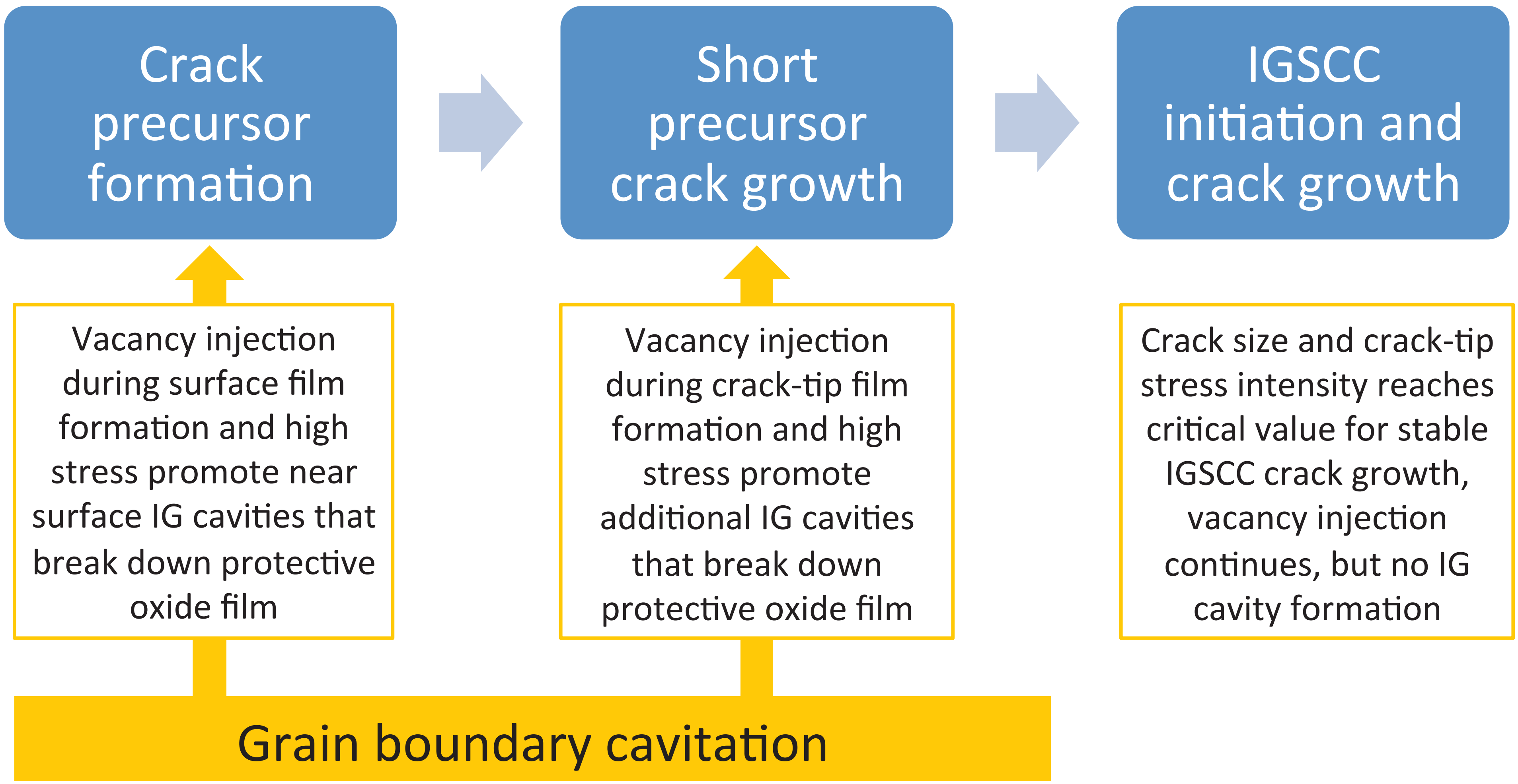




Summary of Alloy 690 Constant Load Test Results

- *Small IG cracks identified on 31%CF specimens from two alloy 690 CRDM heats after constant load testing for 9220 hours in 360°C PWR primary water, no cracking for two other heats in the same condition.*
- *Isolated, very small IG cracks detected on 21%CF specimen from one CRDM heat, no damage on specimens from three other heats.*
- *Extensive grain boundary cavity formation is present associated with shallow IG cracks in the highly cold-worked materials. Selected boundaries show a semi-continuous distribution of cavities extending to the surface. As a result, grain boundary cavity formation appears to be directly involved in the nucleation and growth of small IG cracks.*
- *It remains unclear whether this IG damage evolution will occur in bulk alloy 690 materials with more realistic levels of cold/warm work (e.g., <~15%) and at lower applied stresses. If susceptibility limited to highly cold-worked materials and high stress, abusive surface grinding may produce conditions for SCC initiation/growth through damage layer.*

SCC Initiation in Cold-Worked Alloy 690



Research is continuing to elucidate the role of IG creep and grain boundary cavity formation on crack initiation in Alloy 690 and its weld metals. Key issues will be to establish material, stress and environmental conditions required for IGSCC initiation.

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